AD-A172 278 PLASMA POLYMER COATINGS TO PREVENT PIPELINE CORROSION AND REDUCE FRICTION(U) POLAR MATERIALS INC MARTIN'S CREEK PA G W WALPERT 21 MAY 86 DAAK70-85-C-0100

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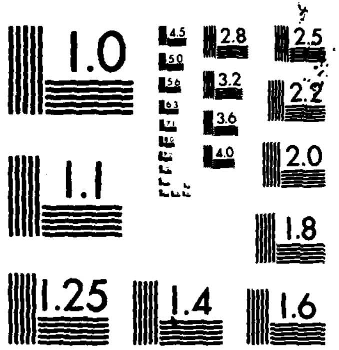

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AD-A172 278

PLASMA POLYMER COATINGS TO PREVENT PIPELINE

CORROSION AND REDUCE FRICTION

CONTRACT #DAAK70-85-C-0100

PRINCIPAL INVESTIGATOR: DR. DEREK SHUTTLEWORTH

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DOD No. 85.1

U.S. DEPARTMENT OF DEFENSE

SMALL BUSINESS INNOVATION RESEARCH PROGRAM
PHASE 1 — FY 1985
PROJECT SUMMARY

Topic No. A-59 Military Department/Agency Army

Name and Address of Proposer

Polar Materials Inc.
Ben Franklin Technology Center
Homer Research, Bldg. F
Bethlehem, PA 18016

Name and Title of Principal Investigator

Dr. Derek Shuttleworth, Director

Proposer's Title*

Plasma Polymer Coatings to Prevent Pipeline Corrosion and Reduce Friction

Technical Abstract* (Limit your abstract to 200 words with no classified or proprietary information/data.)

This program will investigate the preparation of corrosion and friction reduction coatings for pipeline protection prepared by state-of-the-art plasma methods. Plasma techniques can produce coatings of ceramic-like materials with high abrasion resistance. Their chemical unreactivity and uniformity of coverage allows them to function as effective barrier coatings for corrosion protection.

Diagnostic and real-time process control methods will be developed to make the procedures a viable production method.

Anticipated Benefits/Potential Commercial Applications of the Research or Development

This program will have extensive application in corrosion protection of, for example, automotive exhausts and corrosive material pipelines.

List a maximum of 8 Key Words that describe the Project.

Corrosion protection, friction reduction, plasma CVD; plasma polymerization

*Nothing on this page is classified or proprietary information/data

PREFACE

The work reported herein was done under Procurement Instrument Number DAAK70-85-C-0100, August 22, 1985, awarded in response to SBIR proposal for Topic A-59 in Solicitation DOD85-1. The work was done in the laboratories of Polar Materials, Inc. during the period August 22, 1985 to April 22, 1986. The Principal Investigator was Dr. Derek Shuttleworth until March 1, 1986, at which time Dr. Shuttleworth was replaced as Principal Investigator by Dr. George W. Walpert. Other contributors to the project include Dr. Malcolm L. White, Consultant and Staff member at the Sinclair Center for Surface and Coatings Research at Lehigh University, Dr. H. Ronald Thomas, President of PMI, Terry J. Hafford, Paula J. Battavio, and Robert J. Babacz, staff members at PMI.

The Contracting Officer's Representative was Louis H. Tagliaferre, STREB-GP, who was kept informed of progress throughout the project. The contract was originally scheduled to cover the period August 22, 1985 to February 22, 1986, but a 60-day no-cost extension was requested by letter from Dr. Shuttleworth to Mr. Tagliaferre dated February 13, 1986, and acknowledged by telephone by Mr. Tagliaferre.

The SBIR proposal proposed utilizing part of a Post-Doctoral appointment at Lehigh University to use non-uncursive in situ analytical techniques to characterize the plasma polymerization process and seek relationships between plasma parameters and product characteristics. Unfortunately, recruiting efforts for the post-doctorial fellow were not successful, and this portion of the project was not done. The monies budgeted for the post-doctorial effort were diverted to staff members at Lehigh University for product characterization, test development, and testing.

ABSTRACT

Three different materials, thiophene, hexamethyldisiloxane, and hexamethyldisilazane were deposited on mild steel coupons by low temperature radio-frequency plasma polymerization. Statistically designed experiment matrices were utlized to screen a range of operating conditions and optimize coating properties. For two of the materials, a narrower range of conditions was used to produce coating weights of about 400 micrograms per square centimeters, and these coatings were evaluated for corrosion protection and coating physical properties.

Coatings were produced which yielded 256 hours in air-bubbled salt water to 95% corrosion. Uncoated coupons in the same test rusted to 95% in six hours. The best coating was deposited at a rate of 4 micrograms per square centimeter per minute, but reasonably good coatings were produced at coating rates of 20 to 30 micrograms per square centimeter per minute.

The feasibility of corrosion protection coatings using low temperature plasma polymerization has been established by this project, but much process and product development remains for commercialization. Recommendations for continued development work are discussed in this report. Keywords: Ceramic materials; Pipelines; Diagnostic tests; Real time; Process control meth

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INTRODUCTION

This report presents the results of a study to determine the feasibility of using non-equilibrium plasma polymerization to produce coatings for corrosion protection and flow enhancement on the inside of pipes.

The low temperature, or non-equilibrium plasma is currently a procedure of significant importance in semiconductor fabrication (1). This development paves the way for other applications of low temperature plasma by the development of equipment for routine use and by development of some understanding of the principles governing plasma technology. Low temperature plasma, therefore, affords considerable promise as a source of protective coatings for use in a diversity of applications.

An opportunity exists to devise coating procedures in which thin films of high performance material can be deposited at relatively low cost. The challenge and the opportunity is to obtain coatings that are hard, chemically inert, of low permeability to oxygen and water, strongly adhering and low in defects, such as pinholes. Plasma methods afford the capability to produce such films, and represent a higher probability of success than other methods.

A particularly flexible plasma method for the production of thin films is plasma polymerization (2, 3, & 4). In this process, volatile materials are caused to react at essentially room temperature in a gas plasma sustained by high frequency electrical power. The state thus produced is highly reactive, populated by radicals, electrons, ions, metastables and electromagnetic radiation extending into the ultraviolet (5). Energies up to 40 eV are available which is to be compared with typical chemical energies of 3 to 10 eV. Many reaction pathways are opened in an organic gas plasma leading to rearrangement of the starting material and/or deposition of polymeric material on surfaces in the plasma zone (6). Under the appropriate conditions, materials generally not considered to be monomers, eg. benzene, xylene, butane, or even methane gas can be deposited as thin, eg. one micron, coatings which are pinhole-free, conformal, strongly adhering, and crosslinked to the point of being insoluble in common organic solvents.

Such coatings have been produced for the subject study of this report from three starting materials, thiophene, hexamethyldisiloxane and hexamethyldisilazane. Corrosion resistance of the films, and their physical properties have been measured and to some extent these responses related to process conditions.

One of the objectives as stated in the SBIR proposal was to develop coatings for corrosion protection and flow enhancement.

One of the criteria for selecting starting materials was to produce coatings with a range of surface energy, intending to study the effect on resistance to flow. However, informal discussions with Navy investigators on fluid flow projects convinced this author that fluid flow is dominantly controlled by surface topography (and the character of the fluid) and that efforts to enhance flow by surface chemistry offered little probability of success with a cursory effort. In addition, the effort required for the investigation proved to have been underestimated in the SBIR Proposal, and the election was made not to pursue the flow enhancement objective.

EXPERIMENTAL

A. PLASMA EQUIPMENT

A laboratory scale vacuum plasma polymerization reactor of six-inch diameter glass was set up with monomer flow controllers, vacuum gauges, R.F. (radio frequency) power supply, and vacuum pump.

Flow was controlled by Datametrics Type 825 mass flow control valves, Datametrics Type 1511 multi-channel digital display flow controller, and ball-float flow tubes, Air Products E29M150MM1 and E29C150MM2. Pressure was sensed and controlled by a Datametrics Type 1500 digital pressure display and power supply, a Datametrics Type 621 Barocel pressure sensor, an MKS Type 252A exhaust valve controller to maintain constant pressure in a vacuum system, an MKS Type 253A-1-40-1 throttling valve to regulate the removal of gas from a vacuum chamber, and a Varian thermocouple vacuum gauge 804-A. The R.F. power generator, ENI Power Systems Model HF-300, was used with A Heathkit SA2060-A manual impedance matching network. The Varian CD300 mechanical vacuum pump was fitted with a 345 foreline trap filled with molecular sieve material. A Mathis Model TM-100 thickness monitor was set up in the reactor sleeve but eventually malfunctioned, and its use was discontinued.

The cross-shaped glass reactor was equipped with a rotating copper heated stage capable of operating at temperatures up to 400° C. Temperature was initially read on a Eurotherm Model 983 digital indicating controller and then an Omega Model 199KC digital temperature indicator. Both units experienced problems with interference from the pick-up of stray RF power. Temperature was finally read on an Omega Model 7045-K-1500 analog pyrometer. For experimental runs without substrate heating, a stainless steel rotating stage was used. A schematic diagram of the system is shown in Figure 1.

B. MATERIALS

Monomers and gases used included thiophene (Fisher Scientific), hexamethyldisiloxane (HMDSO) and hexamethyldisilazane (HMDSN) (both from Petrarch Systems Inc.), oxygen and argon (both from Air Products). Substrate materials were 0.5 mm thick, smooth finish, steel Type OD Q-Panels, ASTM specification D609.3B.

C. OPERATING PROCEDURES

The Q-Panels were cut into desired sizes, washed with trichloroethylene, and stored in desiccators. Before each run, they were individually wiped with a Kimwipe and trichloroethylene, air dried, weighed, and loaded into the reactor.

Thiophene, HMDSO, and HMDSN were vacuum degassed at liquid nitrogen-temperature. The gas flow from these monomers were manually controlled by flow tubes.

After the substrates were positioned on the rotating, electrically grounded electrode and loaded in the reactor, pressure was lowered to less than 0.3 Torr, and an autotransformer was manually adjusted to the heated rotating electrode to maintain the desired temperature. Substrates were exposed to an argon plasma (gas flow 28 sccm, pressure 500 mTorr, power 100 w) for two minutes to clean the substrate surfaces. Thiophene runs A59A1 to A13, A59B1 to B13, A59C1 to C3, and A59A55 were exposed to the argon plasma followed by an oxygen plasma (gas flow 25 sccm, pressure 500 mTorr, power 100 w) for one minute.

Monomers at a prescribed rate were introduced into the reactor, pressure was set to a prescribed value, R.F. power turned on, adjusted to the prescribed value and forward power optimized with the matching network to maintain a glow discharge plasma. Every 5 minutes during a run, the R.F. power was turned off, the temperature checked, the autotransformer adjusted if necessary to maintain the desired temperature, and the R.F. power turned back on. (Because of R.F. pick-up by the thermocouple leads, the temperature indicator could only be hooked up to the system and the temperature noted when the plasma was off, although power was supplied to the heater via the autotransformer during the entire run.) At the end of a run, the R.F. power, monomer flow, and heater power were turned off. Pressure was pumped down to less than 0.3 Torr. Samples remained in the reactor under vacuum until the temperature fell below 100° C. Then the system was vented, the samples removed from the system, allowed to cool to room temperature, and weighed.

Thiophene Experimental Designs I and II, design orders 2, 3, 5, and 8 were run to different thicknesses on the Mathis Thickness monitor to estimate the correlation between the thickness rate calculated from the actual coating formed and the thickness monitor indication. The remaining design orders in thiophene Designs I, II, and III were run to a thickness of 0.25 kiloangstroms on the monitor to the nearest whole minute. A monitor thickness rate in kiloangstroms per minute was calculated. For samples having the better film qualities, run times were calculated to reach a thickness build-up of 500 micrograms per square centimeter using the thickness rate of the actual coating formed.

D. EXPERIMENTAL DESIGN

Two-level fractional factorial experiments were designed to identify the process variables that most significantly influenced development of a good coating and to begin optimizing a set of process conditions.

Preliminary experiments included full factorial designs for thiophene runs with no temperature control, half factorial designs for thiophene and HMDSO runs with temperature control, and half factorial designs for HMDSN with no temperature control. The process variables studied included conditions varying the radio

frequency, power, pressure, flow, and temperature. The design matrices and ranges for the variables are shown in Tables 1-7.

E. EVALUATION TECHNIQUES

The coating properties measured included weight gain of the coupon, scrape adhesion, tensile adhesion, salt exposure (corrosion resistance), pinhole microscope tests, conductance, and abrasion resistance.

Coating thickness (coating weight) in grams per square centimeter was calculated by the coating weight obtained on the O-Panels divided by the coated area.

A Pacific Scientific, Gardner/Neotec Instrument Division, balanced beam scrape-adhesion and mar tester, SG-8101, fitted with a loop stylus, SG-8102 was used for the scrape adhesion test. The Standard Test Method for Adhesion of Organic Coatings, ASTM D2197-68 (reapproved 1979) Method A Scrape Adhesion, was used with the following modifications to the ASTM procedure: sample size, load increments, number of repeated tests, and detection of adhesion end point using an electrical contact. A coated 2.5 x 2.5 cm square O-Panel was tested with 10-g increments to 100g, 100-g increments to 1 kg, and 1-kg increments to a maximum load of 10 kg. End points were repeated as available sample area permitted. Two indications of failure were reported. Scrape Adhesion 1 was the loading for the first discontinuous indication of electrical contact between the stylus and the substrate, and Scrape Adhesion 2 was the loading for continuous contact.

The tensile adhesion test employed a Sebastian I coating adherence tester (The Quad Group). An epoxy-coated pull stud was clipped to a coated O-Panel, cured at 125° C for 2.5 hours, and cooled to room temperature. The bond strength was determined by the amount of force in psi exerted to pull the stud from the sample. The amount of coating left on the sample was noted.

Salt immersion testing was made by suspending a sample above 5% NaCl solution with the lower half immersed while the solution was bubbled with air. Samples were observed periodically for percentage of surface area oxidized to 95% corrosion.

Deviation from the salt immersion procedure was made for some samples from the HMDSN runs. Samples were submersed into the salt solution bubbled with air and placed flat at the bottom of the container. Observed percentages of surface corrosion were corrected to values relating to the partial immersion testing described above by comparing uncoated substrates evaluated by both methods.

Three pinhole measurements were performed using solutions of neutral $CuSO_4$ ($CuSO_4.5H_2O$, H_2O), acidified $CuSO_4$ ($CuSO_4$, H_2SO_4, H_2O), and Ferroxil ($K_3Fe(CN)_6$, NaCl). A drop of one solution is placed on a coating surface. Using an Olympus zoom binocular

microscope at 10x magnification, the time was noted for the formation of 5 crystals, and then 10 crystals.

The conductance was measured by placing an 0.05-0.1 ml drop of 3% NaCl solution on the coating, immersing a stainless steel wire electrode into the drop, using the substrate as the other electrode, and reading the conductance within ten seconds at 2kHz with an Extech Model 440 conductivity meter.

The abrasion resistance was measured using the falling sand technique (ASTM D968-51). 100 ml volumes of Ottowa silica sand (-20 +30 mesh) were funneled through a 36" long by three-quarter-inch diameter tube and allowed to impinge on the coated substrate, held at a 45° angle, until there was some area of bare metal showing. The total volume of sand to reach this point was recorded.

Contact angle measurements were taken using a Rame-Hart Model A-100 NRL C.A. goniometer and water as the liquid.

Density measurements were approximated by wetting pieces of flaked film in different solutions having a range of densities (HL aqueous series from Cargille Lab). Film flakes having a higher density than the solution sank while flakes having a lower density floated.

Quality of the deposited films were classified as film (F) or powder (P). The visual appearance further described the films as even or rainbow colored, peeled, or flaked. A particular sample could exhibit more than one appearance characteristic at one time, usually in different areas of the substrate.

RESULTS AND DISCUSSION

A. DATA

Three materials were evaluated by plasma polymerization as candidates for pipeline coatings - thiophene, hexamethyldisiloxane and hexamethyldisilazane. Each were selected because of previous personal experience of the investigators, and because of a reference in the literature citing good quality films from these materials. The evaluation was done by coating coupons in a research reactor and measuring film properties and corrosion protection on the coupons.

For each material, a statistically designed 2-level full factorial experiment (8 runs) was run to study the effects of process conditions: gas flow rate, pressure, and RF power input. When the temperature-controlled grounded electrode became available, half-factorial designs of 8 runs were made, including substrate temperature as the fourth variable. For the hexamethyldisilazane runs done at ambient temperature, the four variables in the 8-run half factorial were HMDSN flow, oxygen flow, pressure, and power. Design details are shown in Tables 1-7.

For each of the preliminary factorial designs, a visual evaluation of coating quality was made, coating weight was measured and coating rate determined. Coating properties were measured with a scrape adhesion test, and in some cases, a measure of film integrity was made using a drop of copper sulfate solution (the Priest Test). Based on these results, a second factorial design was run at different ranges for the process variables, or selected runs from the first design were repeated at the same process conditions, but different duration to achieve a targeted coating weight, or for selected runs, process conditions were modified slightly and rerun. A total of eleven "sets" were done, comprising 98 individual coating trials. The printout for all of the run data is shown in the Appendix as Tables 8 to 18.

Tables 19, 20, and 21 present a condensation of the data where coating weight and coating rates have been averaged for all the coupons in each run and test data are tabulated by run rather than by coupon. These tables were used for analysis of effects of process variables and for observations upon which the conclusions were based.

B. GENERAL

The interpretation of the massive amount of data accumulated for this program, the distillation of results and the formulation of conclusions is severely hindered by a large amount of variability. Individual coupons within a run gained different coating weight per unit of area despite the rotating electrode sample holder, replicate runs yielded variable results, and test results on duplicate samples from a given run were not reproducible. Statistical test for significance of the first

order effects of process variables show very few to have significance at the 90% confidence level. The objective to relate coating properties to process conditions was only partially achieved, and to this extent only because the massive amount of data enabled some judgemental conclusions based on consistency of the direction of first order effects.

Since the conclusion of the experimental work on this contract, the research reactor on which this work was done has been equipped with a pulse generator to enable pulsing the RF power input in the range of one millisecond period to one second period. Early indications are that pulsing at about 100 milliseconds results in faster coating rates, better film properties, better consistency and more reproducibility. Any continuation of the investigation should definitely include pulsed power as a parameter for study.

The attempt to understand the source of variability has brought into question the design of the research reactor used for this study. Emphasis on electrical isolation of the RF power electrodes resulted in spaces of stagnant gas which have been observed to be the predominant sources of powder. In a configuration specifically designed to coat the inside of pipe with the active electrode centered in the pipe, the pipe itself as both the counterelectrode and the vacuum chamber wall, and reaction gases flowing between them, there would be no stagnant space. The anticipated effect is for higher quality and more uniform coatings. Continuation of the project should go directly to the coating of lengths of pipe, or at least a research reactor designed to simulate the condition of pipe coating.

The compelling observation to be derived from the data is that a number of runs produced reasonably good corrosion-protection coatings. Table 23 shows the test results of the coatings from 15 of the trials, 9 thiophene and 6 hexamethyldisiloxane. The first row in the table is an arbitrary set of acceptance criteria for this stage of development and the entries within each family of coating are by decreasing corrosion resistance as measured by bubbling salt spray. Those properties deficient with respect to the selection criteria are underlined. Run number A59B93 resulted in coupons meeting all acceptance criteria. There is no consistency among the other runs for properties failing to meet the acceptance criteria. The results listed in this table lead to the conclusions that suitable coating properties are achievable, but that continuation of the development mandates upgrading repeatability, upgrading the design of equipment, procedures, and characterization methods.

C. FILM DENSITY

Flakes of coating from some of the coupons which came from the reactor with the film flaking off were used to determine coating density. For these flakes, densities of 1.4 to 1.7 grams per cubic centimeter were observed. At the conclusion of the

experimentation on this contract, a means to remove good coatings, eg. coating from trials resulting in good salt spray results, had not been devised. It is the consensus of the investigators on this project for plasma polymerized coatings, that there is a relationship between coating density and oxygen and water permeability, and that for coatings as thin as are targeted, film density of about 2 will be required. The identification of process conditions to produce this density without internal stresses that lead to crazing and flaking should be an objective of any continuation of this project.

D. COATING PROPERTIES vs. PROCESS CONDITIONS

The first order effect for each process variable on coating rate and film properties in each experimental design matrix is shown in Table 24. A test for significance was done judgmentally by inspection of computer generated cumulative probability charts for each design matrix. Meaningful effects on the table are underlined. In general, the variability in the process and/or in the measuring procedures obscures any well-defined relationship between process conditions and coating properties. However, some trends are indicated. Increased monomer feed rate (flow) within the range studied in the program produced positive results, ie. faster coating rates, better scrape adhesion results, and lower electrical conductivity. The first order effects of pressure were predominantly not significant, but in many instances, the sign of the effect was opposite the sign of the effect of flow. The important parameter may be residence time, a parameter inversely proportional flow rate and directly proportional to reactor volume and pressure. The concept is interesting, and useful in optimizing trade-off considerations for coating properties vs. economics. When the effects of temperature are judged to have significance, they are mostly negative, ie. detrimental, and the preponderance of temperature effects even where not judged to be significant are negative. If this effect is real, it is contrary to the expected effect based on published literature, which indicates that elevated temperature is required to achieve films with integrity and good corrosion protection. The ability to operate at ambient temperature would obviously be an advantage in a manufacturing process.

The range of process variables utilized for plasma polymerization is limited by regions of the operating conditions that produce powder rather than deposit film. As was observed in paragraph B, entitled General, the design of the research reactor used for this project was a particular offender in producing powder. With a reactor more carefully designed to simulate the process of coating the inside of pipes, the range of the variables studied could be extended and, along with the better reproducibility expected, a better relationship between process conditions and film properties could be obtained.

E. ECONOMICS

The process for coating the inside of pipes was not well enough defined in this program to enable a detailed economic study. Some limiting costs can be estimated. Coating run #A59A89, for instance, provided 216 hours of bubbling salt water protection at a coated weight of 360 micrograms per square centimeter, applied at 10 watts with an electrode area of approximately 650 square centimeters in a 66 minute coating run. For a 21-foot length of 4-inch schedule 40 pipe, assuming 50% material conversion, 50% power efficiency, using chemically pure thiophene at $15 per pound and 5 cents per kilowatt hour for power, the material cost would be 6 cents, and the power cost would be 13 cents. To design a coating plant for reasonable capital cost and reasonable operating cost is an entirely feasible prospect.

RECOMMENDATIONS

The investigation of plasma polymerized coatings for corrosion protection of the inside of pipes should be continued with a multi-pronged effort:

1. The design of a coating reactor to simulate the inside of a pipe. A section of pipe as the reaction chamber would be appropriate, suitably modified with a demountable (vacuum tight) wall segment for obtaining samples of coating for analysis and characterization. The ends of the pipe section would be ground flat for a vacuum tight "O" ring seal to an electrically insulating end plate including the monomer inlet and electrode mount in one end and the vacuum port and electrode mount on the other end. If controlled elevated temperature continues to be of interest, then an electrically heated jacket for the pipe may be provided.

2. Using the reactor described above, and a starting set of process conditions extracted from the work covered in this report, concentrate on achieving reproducibility of the process and the characterization test procedures.

3. With reproducibility achieved, develop the relationship between process conditions and coating performance.

4. Conduct a screening study to select promising material systems for corrosion protection plasma coating. The thiophene, siloxane and silazane starting materials used in this study were selected considering literature references, availability, cost, and safety considerations. Recent work by this author using all-hydrocarbon coatings of multi-component monomers, and two-layer coatings using fluorinated hydrocarbon top layers have shown promise for corrosion protection. Several members of each "family" of coatings should be screened for this application.

5. With the understanding of the process and an optimum materials package from steps 3 and 4 above, develop parameters for the design of a commercial plant and details of the economics of plasma polymerization coating for corrosion protection of fluid-flow pipes.

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APPENDIX

TABLE #	TITLE
1	THIOPHENE EXPERIMENTAL DESIGN I
2	THIOPHENE EXPERIMENTAL DESIGN II
3	THIOPHENE EXPERIMENTAL DESIGN III
4	THIOPHENE EXPERIMENTAL DESIGN IV
5	HMDSO EXPERIMENTAL DESIGN I
6	HMDSN EXPERIMENTAL DESIGN I
7	HMDSN EXPERIMENTAL DESIGN II
8	DATA FOR THIOPHENE DESIGN I
9	DATA FOR THIOPHENE DESIGN II
10	DATA FOR RUNS AT CONDITIONS MODIFIED FROM THIOPHENE DESIGNS I AND II
11	DATA FOR SELECTED RUNS FROM THIOPHENE DESIGNS I AND II AT 500 MICROGRAMS PER SQUARE CENTIMETER
12	DATA FOR THIOPHENE DESIGN III
13	DATA FOR THIOPHENE DESIGN IV
14	DATA FOR THIOPHENE DESIGN III-A AT 500 MICROGRAMS PER SQUARE CENTIMETER
15	DATA FOR SELECTED RUNS FROM THIOPHENE DESIGN IV AT 500 MICROGRAMS PER SQUARE CENTIMETER
16	DATA FOR HMDSO DESIGN I
17	DATA FOR HMDSN DESIGN I
18	DATA FOR HMDSN DESIGN II
19	SUMMARY OF AVERAGED DATA OF RUNS IN THIOPHENE DESIGN I, II, III, AND IV
20	SUMMARY OF AVERAGED DATA OF SELECTED RUNS FROM THIOPHENE DESIGNS III AND IV
21	SUMMARY OF AVERAGED DATA OF RUNS IN HMDSO DESIGN I
22	SUMMARY OF AVERAGED DATA OF RUNS IN HMDSN DESIGNS I AND II
23	PROPERTIES OF THE 15 BEST CORROSION PROTECTION COATINGS
24	FIRST ORDER EFFECTS OF PROCESS VARIABLES

FIG. 1 VACUUM PLASMA POLYMERIZATION REACTOR AND SYSTEM

TABLE 1

THIOPHENE EXPERIMENTAL DESIGN I

Design Order	Run Number	Flow sccm	Press mTorr	Power w	Run Order
1	A59A9	-	-	-	8
2	A59A2	+	-	-	2
3	A59A4	-	+	-	4
4	A59A7	+	+	-	6
5	A59A1	-	-	+	1
6	A59A6	+	-	+	5
7	A59A8	-	+	+	7
8	A59A3	+	+	+	3

Variables	-	+
Flow	2.1	29
Press	250	500
Power	10	50

TABLE 2

THIOPHENE EXPERIMENTAL DESIGN II

Design Order	Run Number	Flow sccm	Press mTorr	Power w	Run Order
1	A59B9	−	−	−	8
2	A59B3	+	−	−	3
3	A59B4	−	+	−	4
4	A59B7	+	+	−	6
5	A59B2	−	−	+	2
6	A59B6	+	−	+	5
7	A59B8	−	+	+	7
8	A59B1	+	+	+	1

Variables	−	+
Flow	2.1	29
Press	500	1000
Power	50	100

Note 1: The following additional thiophene runs were made.

Run Number	Flow sccm	Press mTorr	Power w	Run Order
A59C1	3	250	50	1
A59C2	3	500	100	2

Note 2: The better samples from Thiophene Experimental Designs I and II were repeated to a coating thickness of approximately 0.5mg/sqcm. The flow (−) values were changed from 2.1 to 3 sccm.

TABLE 3

THIOPHENE EXPERIMENTAL DESIGN III

Design Order	Run Number	Power w	Press mTorr	Flow sccm	Temp deg C	Run Order
1	A59A59	−	−	−	−	3
2	A59A57	+	−	−	+	2
3	A59A67	−	+	−	+	8
4	A59A65	+	+	−	−	6
5	A59A66	−	−	+	+	7
6	A59A55	+	−	+	−	1
7	A59A61	−	+	+	−	4
8	A59A63	+	+	+	+	5

Variables	−	+
Power	10	50
Press	250	500
Flow	3	29
Temp	50	150

TABLE 3

THIOPHENE EXPERIMENTAL DESIGN III

Design Order	Run Number	Power w	Press mTorr	Flow sccm	Temp deg C	Run Order
1	A59A59	−	−	−	−	3
2	A59A57	+	−	−	+	2
3	A59A67	−	+	−	+	8
4	A59A65	+	+	−	−	6
5	A59A66	−	−	+	+	7
6	A59A55	+	−	+	−	1
7	A59A61	−	+	+	−	4
8	A59A63	+	+	+	+	5

Variables	−	+
Power	10	50
Press	250	500
Flow	3	29
Temp	50	150

TABLE 4

THIOPHENE EXPERIMENTAL DESIGN IV

Design Order	Run Number	Power w	Press mTorr	Flow sccm	Temp deg C	Run Order
1	A59B72	−	−	−	−	5
2	A59B70	+	−	−	+	3
3	A59B75	−	+	−	+	8
4	A59B71	+	+	−	−	4
5	A59B68	−	−	+	+	1
6	A59B74	+	−	+	−	7
7	A59B69	−	+	+	−	2
8	A59B73	+	+	+	+	6

Variables	−	+
Power	50	100
Press	250	500
Flow	3.9	29
Temp	150	250

Note 1: The better samples from Thiophene Experimental Designs III and IV were repeated to a coating thickness of approximately 500 ug/sqcm.

TABLE 5

HMDSO EXPERIMENTAL DESIGN I

Design Order	Run Number	Power w	Flow sccm	Press mTorr	Temp deg C	Run Order
1	A59S0128	-	-	-	-	8
2	A59S0124	+	-	-	+	4
3	A59S0123	-	+	-	+	3
4	A59S0129	+	+	-	-	9
5	A59S0130	-	-	+	+	10
6	A59S0126	+	-	+	-	6
7	A59S0127	-	+	+	-	7
8	A59S0125	+	+	+	+	5
0	A59S0120, A59S0121 A59S0131, A59S0132	1/2	1/2	1/2	1/2	1,2 11,12

Variables	-	+	1/2
Power	50	200	95
Flow	3.5	30	7,8
Press	350	1000	675
Temp	150	250	200

TABLE 6

HMDSN EXPERIMENTAL DESIGN I

Design Order	Run Number	Power w	Flow HMDSN sccm	Flow O2 sccm	Press mTorr	Run Order
1	86428, 864213	-	-	-	-	8, 13
2	86414	+	-	-	+	4
3	86413	-	+	-	+	3
4	86429	+	+	-	-	9
5	864210	-	-	+	+	10
6	86416	+	-	+	-	6
7	86417	-	+	+	-	7
8	86415	+	+	+	+	5
0	86381, 86382	1/2	1/2	1/2	1/2	1, 2
	864211, 864212					11, 12

Variables	-	+	1/2
Power	50	200	125
Flow HMDSN	3.5	29	8
Flow O2	2	5	3
Press	250	1000	625

Note 1: The following runs were made with the pressure valve left open; ie, minimun pressure for the flow rates with the system vacuum pump.

Run Number	Power w	Flow HMDSN sccm	Flow O2 sccm
864214, 864215	200	3.5	2
864316	200	2.4	2

TABLE 7

HMDSN EXPERIMENTAL DESIGN II

Design Order	Run Number	Power w	Flow HMDSN sccm	Flow O2 sccm	Press mTorr	Run Order
1	86445	–	–	–	–	5
2	86457	+	–	–	+	7
3	86446	–	+	–	+	6
4	864510	+	+	–	–	10
5	86459	–	–	+	+	9
6	86443, 864813	+	–	+	–	3, 13
7	86444	–	+	+	–	4
8	86458	+	+	+	+	8
0	86431, 86432	1/2	1/2	1/2	1/2	1,2
	864511, 864512					11, 12

Variables	–	+	1/2
Power	50	200	125
Flow HMDSN	2.4	3.5	3
Flow O2	0	2	2
Press	175	250	213

TABLE 8

DATA FOR THIOPHENE DESIGN I

Design Order	Run Number	Power w	Press, mT Set	Press, mT Obt	Flow1 Set	Flow2 Obt	Flow sccm	Time min	Sample Number	Weight Gain, g
1	A59A9	10	250	250	10		2.1	12	1	0.0002
								12	2	0.0003
								12	3	0.0002
								12	4	0.0001
2	A59A2	10	250	250	150		29	15	1	0.0032
								15	2	0.0034
								15	3	0.0043
								15	4	0.0038
3	A59A4	10	500	500	10		2.1	40	1	0.0020
								40	2	0.0016
								40	3	0.0015
								40	4	0.0017
4	A59A7	10	500	500	150	135	20	29	1	0.0135
								29	2	0.0150
								29	3	0.0118
								29	4	0.0099
5	A59A1	50	250	250	10		2.1	10	1	0.0008
								10	2	0.0004
								10	3	0.0001
								10	4	0.0002
6	A59A6	50	250	250	150		29	2	1	0.0007
								2	2	0.0008
								2	3	0.0006
								2	4	0.0009
7	A59A8	50	500	500	10		2.1	45	1	0.0005
								45	2	0.0004
								45	3	0.0004
								45	4	0.0004
8	A59A3	50	500	500	150		29	6	1	0.0069
								6	2	0.0062
								6	3	0.0052
								6	4	0.0061

TABLE 8

Wt Rate g/min	Thickness g/sqcm	Thick Rate g/sqcm/min	T-mon rate Ang/min	Qual
1.7E-05	3.2E-05	2.7E-06	21.2	F
2.5E-05	4.8E-05	4.0E-06		F
1.7E-05	3.2E-05	2.7E-06		F
8.3E-06	1.6E-05	1.3E-06		F
2.1E-04	5.1E-04	3.4E-05	22.9	F
2.3E-04	5.4E-04	3.6E-05		F
2.9E-04	6.9E-04	4.6E-05		P
2.5E-04	6.0E-04	4.0E-05		P
5.0E-05	3.2E-04	8.0E-06	5.45	P
4.0E-05	2.6E-04	6.4E-06		P
4.0E-05	2.6E-04	6.4E-06		P
4.2E-05	2.7E-04	6.8E-06		P
4.7E-04	2.1E-03	7.4E-05	8.97	P
5.2E-04	2.4E-03	8.3E-05		P
4.1E-04	1.9E-03	6.5E-05		P
3.4E-04	1.6E-03	5.5E-05		P
8.0E-05	1.3E-04	1.3E-05	18.6	F
4.0E-05	6.4E-05	6.4E-06		F
1.0E-05	1.6E-05	1.6E-06		F
2.0E-05	3.2E-05	3.2E-06		F
3.5E-04	1.1E-04	5.6E-05	364	F
4.0E-04	1.3E-04	6.4E-05		F
3.0E-04	9.6E-05	4.8E-05		F
4.5E-04	1.4E-04	7.2E-05		F
1.1E-05	8.0E-05	1.8E-06	5.91	F
8.9E-06	6.3E-05	1.4E-06		F
8.9E-06	6.3E-05	1.4E-06		F
8.9E-06	6.3E-05	1.4E-06		F
1.2E-03	1.1E-03	1.8E-04	47.3	P
1.0E-03	9.6E-04	1.6E-04		P
8.7E-04	8.4E-04	1.4E-04		P
1.0E-03	9.6E-04	1.6E-04		P

TABLE 8

Comments and Observations
Film
Film
On glass, film
On glass, film
Slight powder
Slight powder
Piece jumped to top electrode during run
On glass, slight powder
Powder; press. drop, took 10 min to line out
Powder; press. drop, took 10 min to line out
On glass, powder; press. drop, took 10 min to line out
On glass, powder; press. drop, took 10 min to line out
Powder; flow dropped to 130
Powder; flow dropped to 130
On glass, powder, some film; flow dropped to 130
On glass, powder, some film; flow dropped to 130
Piece jumped to top electrode during run
Piece jumped to top electrode during run
On glass, film
On glass, film
Film, small amt powder
Film, small amt powder
On glass, film, small amt powder
On glass, film, small amt powder
Film, small amt powder
Film, small amt powder
On glass, film
On glass, film
Loose powder
Loose powder
On glass, loose powder
On glass, loose powder

TABLE 8

Scratch 1	Scratch 2	Pull Test	Acid 5cry	CuSO4 10cry	Neut 5cry	CuSO4 10cry	Ferroxil 5cry	Ferroxil 10cry
0	0							
			0	0	0.1	0.2	>0.1	
50	100							
1000	5000		1.8	3.5	0.6		0.2	0.5
500	1500	20						
100	2000		>0.5		2.5		>0.4	
500	5000		0.2	0.3	2.2		>0.2	
100	900	1140						
100	2000							
200	500	30						
100	400							
200	400		0.2		0.1	0.2	0	0
0	700							
1000	5000		0.2	0.4	>1.2		0.1	0.2
0	4000							
0	5000							
0	800		0.2	0.5	>0.4		>0.2	
0	600							
10	1000							
50	900							

Note for microscope tests time to 5 and 10 crystals is in min and > means time to less than 5 crystals.

TABLE 8

Notes

Ac $CuSO_4$ formed 500+ crystals in 27 sec

Scratch test 2 questionable
Scratch test 2 questionable

Scratch test 2 questionable

Ferroxil formed 50+ crystals in 10 sec

Stud not affix to coating for Pull test
Scratch test 2 questionable
Scratch test 2 questionable
Scratch test 2 questionable
Stud not affix to coating for Pull test

TABLE 9

DATA FOR THIOPHENE DESIGN II

Design	Run	Power	Press. mT		Flow1	Flow2	Flow	Time	Sample	Weight
Order	Number	W	Set	Obt	Set	Obt	sccm	min	Number	Gain, g
1	A59B9	50	500	500	10		2.1	74	1	0.0006
								74	2	0.0007
								74	3	0.0012
								74	4	0.0005
2	A59B3	50	500	500	150		29	4	1	0.0041
								4	2	0.0039
								4	3	0.0037
								4	4	0.0036
3	A59B4	50	1000	510	10		2.1	17	1	0.0012
								17	2	0.0012
								17	3	0.0009
								17	4	0.0012
4	A59B7	50	1000	1000	140	135	20	7	1	0.0067
								7	2	0.0063
								7	3	0.0046
								7	4	0.0046
5	A59B2	100	500	500	10		2.1	25	1	0.0004
								25	2	0.0005
								25	3	0.0003
								25	4	0.0003
6	A59B6	100	500	500	150		29	2	1	0.0011
								2	2	0.0011
								2	3	0.0010
								2	4	0.0011
7	A59B8	100	1000	<1000	10		2.1	42	1	0.0007
								42	2	0.0006
								42	3	0.0018
								42	4	0.0022
8	A59B1	100	1000	1000	150		29	3	1	0.0038
								3	2	0.0038
								3	3	0.0034
								3	4	0.0034

TABLE 9

Wt Rate g/min	Thickness g/sccm	Thick Rate g/sccm/min	T-mon rate Ang/min	Qual
8.1E-06	9.6E-05	1.3E-06	3.38	F
9.5E-06	1.1E-04	1.5E-06		F
1.6E-05	1.9E-04	2.6E-06		F
6.8E-06	8.1E-05	1.1E-06		F
1.0E-03	6.4E-04	1.6E-04	76.8	P
9.8E-04	6.4E-04	1.6E-04		P
9.2E-04	6.0E-04	1.5E-04		P
9.0E-04	5.6E-04	1.4E-04		P
7.1E-05	1.9E-04	1.1E-05	14.5	P
7.1E-05	1.9E-04	1.1E-05		P
5.3E-05	1.4E-04	8.5E-06		P
7.1E-05	1.9E-04	1.1E-05		P
9.6E-04	1.0E-03	1.5E-04	38.6	P
9.0E-04	1.0E-03	1.4E-04		P
6.6E-04	7.0E-04	1.0E-04		P
6.9E-04	7.7E-04	1.1E-04		P
1.6E-05	6.5E-05	2.6E-06	9.72	F
2.0E-05	8.0E-05	3.2E-06		F
1.2E-05	4.8E-05	1.9E-06		F
1.2E-05	4.8E-05	1.9E-06		F
5.5E-04	1.8E-04	8.8E-05	172	F
5.5E-04	1.8E-04	8.8E-05		F
5.0E-04	1.6E-04	8.0E-05		F
5.5E-04	1.8E-04	8.8E-05		F
1.7E-05	1.1E-04	2.7E-06	5.81	F
1.4E-05	9.7E-05	2.3E-06		F
4.3E-05	2.9E-04	6.9E-06		F
5.2E-05	3.5E-04	8.4E-06		F
1.3E-03	6.0E-04	2.0E-04	116	P
1.3E-03	6.0E-04	2.0E-04		P
1.1E-03	5.4E-04	1.8E-04		P
1.1E-03	5.4E-04	1.8E-04		P

TABLE 9

Comments and Observations
Film
Film
On glass, film
On glass, film
Partial powder
Partial powder
On glass, partial powder
On glass, partial powder
Powder
Powder
On glass, powder
On glass, powder
Thick powder; flow unstable
Thick powder; flow unstable
On glass, thick powder; flow unstable
On glass, thick powder; flow unstable
Film
Film
On glass, film
On glass, film
Film, small amt powder
Film, small amt powder
On glass, film
On glass, film
Film, small amt powder; press, took 20 min to line out
Film, small amt powder; press, took 20 min to line out
On glass, film; press, took 20 min to line out
On glass, film; press, took 20 min to line out
Powder
Powder
On glass, powder
On glass, powder

TABLE 9

Scratch 1	Scratch 2	Pull Test	Acid CuSO4 5cry	Acid CuSO4 10cry	Neut CuSO4 5cry	Neut CuSO4 10cry	Ferroxil 5cry	Ferroxil 10cry
			0.2	0.4	0.1	0.2	0.1	
0	500							
0	300							
100	200							
100	600		0.1	0.2	0.7		0.1	0.2
100	300							
100	200		0.1	0.2	0.3		0	0.1
10	1000							
			0.2		>0.3		>0.2	
10	150							
0	10							
0	200							
			0.1	0.2	0.2	0.3	0.1	

TABLE 9

Notes

Stud not affix to coating for Pull test

Stud not affix to coating for Pull test

Scratch test 2 questionable

TABLE 10
DATA FOR RUNS AT CONDITIONS MODIFIED FROM THIOPHENE DESIGNS I AND II

Design Order	Run Number	Power w	Press. mT Set	Press. mT Obt	Flow1 Set	Flow2 Obt	Flow sccm	Time min	Sample Number	Weight Gain, g
5	A5901	50	250	150	15		3	6	1	0.0005
								6	2	0.0005
								6	3	0.0004
								6	4	0.0003
5	A5902	100	500	450	15		3	10	1	0.0004
								10	2	0.0002
								10	3	0.0001
								10	4	0.0005

TABLE 10

wt Rate g/min	Thickness g/sccm	Thick Rate g/sccm/min	T-mon rate Ang/min	Qual
6.3E-05	7.8E-05	1.3E-05	43.7	F
8.3E-05	7.8E-05	1.3E-05		F
6.7E-05	6.6E-05	1.1E-05		F
8.3E-05	7.8E-05	1.3E-05		F
4.0E-05	6.4E-05	6.4E-06	25.6	F
2.0E-05	3.2E-05	3.2E-06		F
3.0E-05	4.8E-05	4.8E-06		F
5.0E-05	8.0E-05	8.0E-06		F

TABLE 10

Comments and Observations
Film: press. drop to 120
Film: press. drop to 120
On glass. film: press. drop to 120
On glass. film: press. drop to 120
mostly film: press. drop, took 6 min to line out
mostly film: press. drop, took 6 min to line out
On glass. mostly film: press. drop. took 6 min to line out
On glass. mostly film: press. drop. took 6 min to line out

TABLE 10

Scratch 1	Scratch 2	Pull Test	Acid 5cry	CuSO4 10cry	Neut 5cry	CuSO4 10crv	Ferroxil 5crv	Ferroxil 10crv
0	100		0.2	0.3	0.2	0.3	>0.7	
0	500							
10	500							
80	500							
0	900							
10	900		1.8		3.0		>0.3	
20	1000							

TABLE 10

Notes
Scratch test 2 questionable
Scratch test 2 questionable
Stud not affix to coating for Pull test
Stud not affix to coating for Pull test

TABLE 11
DATA FOR SELECTED RUNS FROM THIOPHENE DESIGNS I AND II AT 500 MICROGRAMS PER SQ. CENTIMETER

Design Order	Run Number	Power w	Press. mT Set	Obt	Flow1 Set	Flow2 Obt	Flow sccm	Time min	Temp degC	Sample Number	Weight Gain, g
1	A59A13	10	250	220	15		3	120	<82	1	0.0034
								120		2	0.0032
								120		3	0.0033
								120		4	0.0033
								120		5	0.0045
								120		6	0.0039
5	A59A11	50	250	153	15		3	120	>68	1	0.0011
								120		2	0.0022
								120		3	0.0031
								120		4	0.0027
								120		5	0.0037
								120		6	0.0039
5	A59C3	50	250	250	15		3	38	<82	1	0.0010
								38		2	0.0010
								38		3	0.0012
								38		4	0.0010
								38		5	0.0011
								38		6	0.0012
6	A59A10	50	250	250	150		29	12	<82	1	0.0034
								12		2	0.0040
								12		3	0.0037
								12		4	0.0036
								12		5	0.0052
								12		6	0.0056
7	A59C4	50	500	400	15		3	120	>82	1	0.0025
								120		2	0.0022
								120		3	0.0024
								120		4	0.0024
								120		5	0.0037
								120		6	0.0038
1	A59B13	50	500	310	15		3	120	>127	1	0.0036
								120		2	0.0034
								120		3	0.0038
								120		4	0.0033
								120		5	0.0045
								120		6	0.0048
5	A59B11	100	500	500	15		3	124	>138	1	0.0018
								124		2	0.0022
								124		3	0.0020
								124		4	0.0018
								124		5	0.0028
								124		6	0.0023
6	A59B10	100	500	500	150	143	25	13	?	1	0.0066
								13		2	0.0069
								13		3	0.0075
								13		4	0.0073
								13		5	0.0069
								13		6	0.0101
7	A59B12	100	1000	1000	15		3	120	>138	1	0.0039
								120		2	0.0042
								120		3	0.0047
								120		4	0.0040
								120		5	0.0055
								120		6	0.0060

TABLE 11

wt Rate g/min	Thickness g/sccm	Thick Rate g/sccm/min	T-mon rate Ang/min	Qual
2.8E-05	5.4E-04	4.5E-06	68.0	F
2.7E-05	5.2E-04	4.3E-06		F
2.8E-05	5.3E-04	4.4E-06		F
2.8E-05	5.3E-04	4.4E-06		F
3.8E-05	4.7E-04	3.9E-06		F
3.2E-05	4.1E-04	3.4E-06		F
1.6E-05	3.0E-04	2.5E-06	62.7	F
1.8E-05	3.5E-04	2.9E-06		F
1.8E-05	3.4E-04	2.8E-06		F
2.2E-05	4.3E-04	3.6E-06		F
3.1E-05	3.8E-04	3.2E-06		F
3.2E-05	4.1E-04	3.4E-06		F
2.6E-05	1.6E-04	4.2E-06	20.2	F
2.6E-05	1.6E-04	4.2E-06		F
3.2E-05	1.9E-04	5.0E-06		F
2.6E-05	1.6E-04	4.2E-06		F
2.6E-05	1.6E-04	4.2E-06		F
3.2E-05	1.9E-04	5.0E-06		F
2.8E-04	5.4E-04	4.5E-05	156	F
3.3E-04	6.4E-04	5.3E-05		F
3.1E-04	5.9E-04	4.9E-05		F
3.2E-04	6.1E-04	5.1E-05		F
4.3E-04	5.4E-04	4.5E-05		F
4.7E-04	5.8E-04	4.8E-05		F
2.1E-05	4.0E-04	3.3E-06	39.6	F
1.8E-05	3.5E-04	2.9E-06		F
2.0E-05	3.8E-04	3.2E-06		F
2.0E-05	3.8E-04	3.2E-06		F
3.1E-05	3.8E-04	3.2E-06		F
3.2E-05	4.0E-04	3.3E-06		F
3.0E-05	5.8E-04	4.8E-06	57.0	F
2.8E-05	5.4E-04	4.5E-06		F
3.2E-05	6.1E-04	5.1E-06		F
2.8E-05	5.3E-04	4.4E-06		F
3.8E-05	4.7E-04	3.9E-06		F
4.0E-05	4.9E-04	4.1E-06		F
1.5E-05	2.9E-04	2.3E-06	9.90	F
1.8E-05	3.5E-04	2.8E-06		F
1.6E-05	3.2E-04	2.6E-06		F
1.5E-05	2.9E-04	2.3E-06		F
2.3E-05	2.9E-04	2.3E-06		F
1.9E-05	2.4E-04	1.9E-06		F
5.1E-04	1.1E-03	8.1E-05	125	p
5.3E-04	1.1E-03	8.5E-05		p
5.8E-04	1.2E-03	9.2E-05		p
5.6E-04	1.2E-03	9.0E-05		p
5.3E-04	7.2E-04	5.5E-05		p
7.8E-04	1.0E-03	8.0E-05		p
3.2E-05	6.2E-04	5.2E-06	31.2	p
3.5E-05	6.7E-04	5.6E-06		p
3.9E-05	7.6E-04	6.3E-06		p
3.3E-05	6.4E-04	5.3E-06		p
4.6E-05	5.6E-04	4.7E-06		p
5.0E-05	6.2E-04	5.2E-06		p

TABLE 11

Comments and Observations

Film: slow press. drop (250-206-197): flow unstable
Film: slow press. drop (250-206-197): flow unstable
Film: slow press. drop (250-206-197): flow unstable
Film: slow press. drop (250-206-197): flow unstable
Film: slow press. drop (250-206-197): flow unstable
Film: slow press. drop (250-206-197): flow unstable
Film: press. drop; flow unstable
Film: press. drop: flow unstable
Film: press. drop: flow unstable
Film: press. drop: flow unstable
Film: press. drop: flow unstable
Film: press. drop: flow unstable
Film rainbow with gold hue
Film rainbow with gold hue
Film rainbow with gold hue
Film rainbow with gold hue
Film rainbow with gold hue
Film rainbow with gold hue
Film rainbow with gold hue, slightly powdery
Film rainbow with gold hue, slightly powdery
Film rainbow with gold hue, slightly powdery
Film rainbow with gold hue, slightly powdery
Film rainbow with gold hue, slightly powdery
Film rainbow with gold hue, slightly powdery
Film: some brown powder; flow unstable; press. drop to 206
Film: some brown powder; flow unstable; press. drop to 206
Film: some brown powder; flow unstable; press. drop to 206
Film: some brown powder; flow unstable; press. drop to 206
Film: some brown powder; flow unstable; press. drop to 206
Film: some brown powder; flow unstable; press. drop to 206
Part film + powder; press. + RF power reverse unstable
Part film + powder; press. + RF power reverse unstable
Part film + powder; press. + RF power reverse unstable
Part film + powder; press. + RF power reverse unstable
Part film + powder; press. + RF power reverse unstable
Part film + powder; press. + RF power reverse unstable
Film, small amt brown powder
Film, small amt brown powder
Film, small amt brown powder
Film, small amt brown powder
Film, small amt brown powder
Film, small amt brown powder
Thick powder; flow unstable
Thick powder: flow unstable
Thick powder; flow unstable
Thick powder: flow unstable
Thick powder: flow unstable; sample dropped
Thick powder: flow unstable
Deep brown powder, some film
Deep brown powder, some film
Deep brown powder, some film
Deep brown powder, some film
Deep brown powder, some film
Deep brown powder, some film

TABLE 11

Scratch 1	Scratch 2	Pull Test	Acid 5crv	CuSO4 10crv	Neut 5crv	CuSO4 10crv	Ferroxil 5crv	Ferroxil 10crv	Salt
2000	5000								
			>0.7		>4.5		>5.9		
2000	5000	0							
									1
							>0.8		
3000	6000	0							
900	10000		>0.3		>2.6				
									1
500	5000								
500	5000								
			0.2	0.4	0.1	0.2	>2.2		
									48
10	6000								
500	5000	0							
400	5000	10							
			1.0	1.3					
900	2000		0.5		0.7	1.4	>0.2		
									1
1000	6000								
500	7000								
			3.3		2.6		>6.0		
									1
	10000	10							
	7000								
			1.1		2.8		>4.0		
									1
50	5000								
			1.8		1.8		>1.7		
200	4000	10							
									48
	10000								
1000	6000	810							
			2.9		0.6	1.2	>3.5		
									5

TABLE 11

Notes

Ac CuSO4 & Ferr wrinkled coating; Ferr formed 10 blob

Coating wrinkled 40 min into SS test
Ferroxil wrinkles coating

Ac CuSO4 eats coating in 2 min

Coating flaked off 40 min into SS test
Scratch tests 1 & 2 questionable
Scratch tests questionable; stud not affix to coating

20% rust after 1 da

Scratch test 2 questionable

Scratch test 2 questionable
Ac CuSO4 wrinkled coating

Coating wrinkled 40 min into SS test
Stud not affix to coating for Pull test

Coating wrinkled and flaked 40 min into SS test
Scratch test questionable
Scratch test questionable

Ferr formed white then 500+ yellow crys in 23 min
Most of coating flaked 1 h into SS test

30% rust after 1 da

Scratch tests questionable
Scratch tests questionable

Coating flaked off 5 h into SS test

TABLE 12

DATA FOR THIOPHENE DESIGN III

Design Order	Run Number	Power w	Press, mT set	Press, mT obs	Flow set	Flow obs	Flow sccm	Temp, deg C set	Temp, deg C obs	Time min
1	A59A59	10	250	248	15	15	3	50	56	10 10
2	A59A57	50	250	248	15	14.6	2.9	150	147	3 3
3	A59A67	10	500	485	15	15	3	150	146	8 8
4	A59A65	50	500		15	15	3	50	58	4 4
5	A59A66	10	250	249	150	150	29	150	148	5 5
6	A59A55	50	250	249	150	150	29	50	57	3 3
7	A59A61	20	500		150	150	29	50	50	13 13
8	A59A63	50	500		150	148	28	150	160	1 3

TABLE 12

Sample Number	Weight Gain, g	Wt Rate g/min	Thickness g/sccm	Thick Rate g/sccm/min	T-mon Rate Ang/min	Qual ity
1	0.0002	2.0E-05	3.2E-05	3.2E-06	31.9	F
2	0.0002	2.0E-05	3.2E-05	3.2E-06	31.9	F
1	0.0002	6.7E-05	5.4E-05	1.8E-05	98.3	F
2	0.0001	3.3E-05	2.8E-05	9.2E-06	98.3	F
1	0.0001	1.2E-05	1.6E-05	2.0E-06	32.9	F
2	-0.0002				32.9	F
1	0.0002	5.0E-05	3.2E-05	8.0E-06	67.0	F
2	0.0001	2.5E-05	1.6E-05	4.0E-06	67.0	F
1	0.0001	2.0E-05	1.6E-05	3.2E-06	58.2	F
2	0.0001	2.0E-05	1.6E-05	3.2E-06	58.2	F
1	0.0005	1.7E-04	8.1E-05	2.7E-05	245	F
2	0.0009	3.0E-04	1.4E-04	4.8E-05	245	F
1	0.0002	1.5E-05	3.2E-05	2.5E-06	22.7	F
2	0.0004	3.1E-05	6.4E-05	4.9E-06	22.7	F
1	0.0011	3.7E-04	1.8E-04	6.0E-05	503	P
2	0.0009	3.0E-04	1.4E-04	4.8E-05	503	P

TABLE 12

Comments and Observations	Scratch 1	Scratch 2
	4	
Eliminate oxygen pretreat: use	4	
thin copper plate	4	
Press drop to 440	4	
Press 336 during run(?); sample 2. cloudy. some powder	4	400
Flow needed adjustment	4	10
	20	700
Unable to get power at 10 w: film looked cloudy	4	10
Gold film with thin layer gold powder over it	10	500

TABLE 13

DATA FOR THIOPHENE DESIGN IV

Design Order	Run Number	Power W	Press. mT set	Press. mT obs	Flow set	Flow obs	Flow sccm	Temp. deg C set	Temp. deg C obs	Time min
1	A59B72	50	250	249	15	15	3	150	146	40 40
2	A59B70	100	250	249	15	15	3	250	254	22 22
3	A59B75	50	500	500	15	15	3	250	246	35 35
4	A59B71	100	500	500	15	15	3	150	156	45 45
5	A59B68	50	250	250	150	150	29	250	240	12 12
6	A59B74	100	250	253	150	150	29	150	155	15 11
7	A59B69	50	500	500	150	150	29	150	140	12 15
8	A59B73	100	500	498	150	150	29	250	261	15 15

TABLE 13

Sample Number	Weight Gain, g	Wt Rate g/min	Thickness g/sccm	Thick Rate g/sccm/min	Quality
1	0.0004	1.0E-05	6.4E-05	1.6E-06	F
2	0.0004	1.0E-05	6.4E-05	1.6E-06	F
1	0.0001	4.5E-06	1.6E-05	7.3E-07	BF
2	0.0000				BF
1	0.0000				F
2	0.0003	8.6E-06	4.9E-05	1.4E-06	F
1	0.0002	4.4E-06	3.2E-05	7.1E-07	F
2	0.0003	6.7E-06	5.0E-05	1.1E-06	F
1	0.0012	8.0E-05	2.0E-04	1.3E-05	F
2	0.0011	7.3E-05	1.8E-04	1.2E-05	F
1	0.0015	1.0E-04	2.4E-04	1.6E-05	F
2	0.0017	1.1E-04	2.7E-04	1.8E-05	F
1	0.0048	3.2E-04	7.7E-04	5.1E-05	P
2	0.0051	3.4E-04	8.1E-04	5.4E-05	P
1	0.0015	1.0E-04	2.4E-04	1.6E-05	F
2	0.0021	1.4E-04	3.3E-04	2.2E-05	F

TABLE 13

Comments and Observations	Scratch 1	Scratch 2	Notes and Observations
Rotate platform by hand: press fluctuated	4	10	
Blue hew (oxidation?); press fluc.: rotate platform by hand	4		
Press fluctuated	4	50	
Rotate platform by hand	4	20	
	40	2500	
Press fluctuated	250	7000	
Gold film under powder	2000	4500	Scratch tests questionable
	300	6000	
Press fluctuated	70	3000	

TABLE 14
DATA FOR THIOPHENE DESIGN III-A
AT 500 MICROGRAMS PER SQ. CENTIMETER

Design Order	Run Number	Power w	Press, mT set	Press, mT obs	Flow set	Flow obs	Flow sccm	Temp, deg C set	Temp, deg C obs	Time min
1	A59A86	10	250	248	15	15	3	50	54	120
										120
										120
										120
										120
										120
2	A59A85	50	250	248	15	15	3	150	149	36
										36
										36
										36
										36
										36
3	A59A90	10	500	499	15	15	3	150	150	120
										120
										120
										120
										120
										120
4	A59A88	50	500	498	15	15	3	50	76	83
										83
										83
										83
										83
										83
5	A59A89	10	250	248	150	148	28	150	148	66
										66
										66
										66
										66
										66
6	A59A84	50	250	248	150	150	29	50	68	14
										14
										14
										14
										14
										14
7	A59A87	20	500	499	150	150	29	50	47	125
										125
										125
										125
										125
										125

TABLE 14

Sample Number	Weight Gain, g	Wt Rate g/min	Thickness g/sccm	Thick Rate g/sccm/min	T-mon Rate Ang/min	Quality
1	0.0038	3.2E-05	6.1E-04	5.1E-06		F
2	0.0039	3.2E-05	6.2E-04	5.2E-06		F
3	0.0037	3.1E-05	5.9E-04	4.9E-06		F
4	0.0038	3.2E-05	6.1E-04	5.1E-06		F
5	0.0053	4.4E-05	5.5E-04	4.6E-06		F
6	0.0057	4.8E-05	5.9E-04	4.9E-06		F
1	0.0004	1.1E-05	6.5E-05	1.8E-06		F
2	0.0005	1.4E-05	7.9E-05	2.2E-06		F
3	0.0003	8.3E-06	4.7E-05	1.3E-06		F
4	0.0003	8.3E-06	4.7E-05	1.3E-06		F
5	0.0004	1.1E-05	4.3E-05	1.2E-06		F
6	0.0006	1.7E-05	6.1E-05	1.7E-06		F
1	0.0025	2.1E-05	4.0E-04	3.3E-06		F
2	0.0024	2.0E-05	3.8E-04	3.2E-06		F
3	0.0023	1.9E-05	3.7E-04	3.1E-06		F
4	0.0027	2.2E-05	4.3E-04	3.6E-06		F
5	0.0038	3.2E-05	4.0E-04	3.3E-06		F
6	0.0033	2.8E-05	3.4E-04	2.8E-06		F
1	0.0024	2.9E-05	3.8E-04	4.6E-06		F
2	0.0020	2.4E-05	3.2E-04	3.9E-06		F
3	0.0022	2.7E-05	3.5E-04	4.2E-06		F
4	0.0023	2.8E-05	3.7E-04	4.4E-06		F
5	0.0036	4.3E-05	3.7E-04	4.5E-06		F
6	0.0035	4.2E-05	3.7E-04	4.4E-06		F
1	0.0024	3.6E-05	3.8E-04	5.8E-06		F
2	0.0016	2.4E-05	2.6E-04	3.9E-06		F
3	0.0020	3.0E-05	3.2E-04	4.8E-06		F
4	0.0021	3.2E-05	3.4E-04	5.1E-06		F
5	0.0043	6.5E-05	4.4E-04	6.7E-06		F
6	0.0040	6.1E-05	4.2E-04	6.3E-06		F
1	0.0027	1.9E-04	4.3E-04	3.1E-05		F
2	0.0027	1.9E-04	4.3E-04	3.1E-05		F
3	0.0028	2.0E-04	4.5E-04	3.2E-05		F
4	0.0027	1.9E-04	4.3E-04	3.1E-05		F
5	0.0040	2.9E-04	4.2E-04	3.0E-05		F
6	0.0036	2.6E-04	3.6E-04	2.6E-05		F
1	0.0035	2.8E-05	5.6E-04	4.5E-06		P
2	0.0028	2.2E-05	4.5E-04	3.6E-06		P
3	0.0037	3.0E-05	5.9E-04	4.7E-06		P
4	0.0037	3.0E-05	5.9E-04	4.7E-06		P
5	0.0066	5.3E-05	6.8E-04	5.4E-06		P
6	0.0049	3.9E-05	5.0E-04	4.0E-06		P

TABLE 14

Comments and Observations
Press fluctuated
Press drop to 220, rose slowly and fluctuated
Press drop to 448, rose slowly and fluctuated
Press drop to 364, rose slowly and fluctuated
Press fluctuated; thiophene ran out
Press fluctuated
Press fluctuated; coatings, fine layer powder over film

TABLE 14

Scratch 1	Scratch 2	Pull psi	Acid 5cry	CuSO4 10cry	Neut 5cry	CuSO4 10cry	Ferroxil 5cry	Ferroxil 10cry	Salt Spray, h	[illegible] uS
500	2000									
700	2000	0								
			0.3	0.6	0.4	1.2	4.9	9.8		
		0								
										1
									122	
4	20	300								
			0	0	0	0	2.5	3.7		
			0	0	0	0	0.3	0.6		
										[illegible]
									42	
100	10000									
900	6000	1280								
			6.1	12.4		19.1	7.4	17.4		
									102	
										3
700	2000									
700	3000	1060								
			0.4	1.2	2.0	6.9	20.0	20.2		
										1
									243	
10	5000									
		1070								
			0.2	0.6	1.9	3.8	0.8	23.7		
										3
									216	
400	6000									
800	7000	160								
			5.4	9.8	45.0	90.0	0.7	1.6		
									72	
										1
90	3000	150								
			4.2	9.0	11.7	23.2	1.2	4.5		
									120	
										1

TABLE 14

Abrasion ml. Sand
200
700
300
200
400
200
100

TABLE 14

Notes and
Observations

Pull test. stud came off when clip removed

Pull test. stud came off when loading in tester

Salt test failed 126 h @95%
Pull test (in excess of). some film left
4c $CuSO_4$. 500+ cry (6sec); N $CuSO_4$. same (15 sec)

Salt test failed 48 h @95%
Scratch)10000 (max)
Pull test (in excess of).) half film left
N $CuSO_4$. needle-like crys form around edges

Salt test failed 168 h @95% (test interrupted)

Pull test (in excess of). some film left
Ferroxil begins cry @ 19 min (cry around edges)

Salt test failed 245 h @95% (test interrupted)

Pull test (in excess of), some film left
Ferroxil begins cry @ 22 min (cry around edges)

Salt test failed 216 h @95% (test interrupted)

Pull test (in excess of). half film left

N $CuSO_4$ 3 cry (27 min)
Salt test failed 72 h @95%

Pull test (in excess of). some film left

Salt test failed 120 h @95%

TABLE 15
DATA FOR SELECTED RUNS FROM THIOPHENE DESIGN IV AT 500 MICROGRAMS PER SQ. CENTIMETER

Design Order	Run Number	Power W.	Press. mT set	Press. mT obs	Flow set	Flow obs	Flow sccm	Temp. deg C set	Temp. deg C obs	Time min
1	A59B33	50	250	230	15	129	20	150	155	120
										120
										120
										120
										120
										120
3	A59B36	50	500	500	15	15	3	250	248	120
										120
										120
										120
										120
										120
4	A59B92	100	500	71	15	32	2	150	134	52
										52
										52
										52
										52
										52
5	A59B91	50	250	250	150	150	29	250	236	42
										42
										42
										42
										42
										42
6	A59B95	100	250	250	150	150	29	150	151	29
										29
										29
										29
										29
										29
6	A59B94	100	500	498	150	150	29	250	252	26
										26
										26
										26
										26
										26

TABLE 15

Sample Number	Weight Gain, g	Wt Rate g/min	Thickness g/sqcm	Thick Rate g/sqcm/min	T-mon Rate Amp/min	Qual ity
1	0.0166	1.4E-04	2.6E-03	2.2E-05		F
2	0.0144	1.2E-04	2.3E-03	1.9E-05		F
3	0.0165	1.4E-04	2.6E-03	2.2E-05		F
4	0.0154	1.3E-04	2.4E-03	2.0E-05		F
5	0.0234	2.0E-04	2.4E-03	2.0E-05		F
6	0.0279	2.3E-04	2.9E-03 2.5	2.4E-05 2.1		F
1	0.0010	8.3E-06	1.6E-04	1.3E-06	55.0	F
2	0.0007	5.8E-06	1.1E-04	9.3E-07	59.0	F
3	0.0010	8.3E-06	1.6E-04	1.3E-06	59.0	F
4	0.0007	5.8E-06	1.1E-04	9.3E-07	59.0	F
5	0.0013	1.1E-05	1.3E-04	1.1E-06	55.0	F
6	0.0015	1.2E-05	1.6E-04	1.3E-06	59.0	F
1	0.0029	5.6E-05	4.6E-04	6.9E-06		F
2	0.0038	7.3E-05	6.2E-04	1.2E-05		F
3	0.0030	5.8E-05	4.8E-04	9.2E-06		F
4	0.0033	6.3E-05	5.2E-04	1.0E-05		F
5	0.0039	7.5E-05	4.0E-04	7.7E-06		F
6	0.0045	8.7E-05	4.6E-04	8.9E-06		F
1	0.0023	5.5E-05	3.7E-04	8.8E-06		F
2	0.0023	5.5E-05	3.7E-04	8.8E-06		F
3	0.0026	6.2E-05	4.2E-04	9.9E-06		F
4	0.0022	5.2E-05	3.5E-04	8.4E-06		F
5	0.0054	1.3E-04	5.5E-04	1.3E-05		F
6	0.0029	6.9E-05	3.0E-04	7.1E-06		F
1	0.0039	1.3E-04	6.4E-04	2.2E-05		F
2	0.0045	1.6E-04	7.2E-04	2.5E-05		F
3	0.0038	1.3E-04	6.1E-04	2.1E-05		F
4	0.0039	1.3E-04	6.4E-04	2.2E-05		F
5	0.0082	2.8E-04	8.4E-04	2.9E-05		F
6	0.0047	1.6E-04	4.9E-04	1.7E-05		F
1	0.0037	1.4E-04	6.0E-04	2.3E-05		F
2	0.0051	2.0E-04	8.1E-04	3.1E-05		F
3	0.0033	1.3E-04	5.2E-04	2.0E-05		F
4	0.0036	1.4E-04	5.7E-04	2.2E-05		F
5	0.0059	2.3E-04	6.0E-04	2.3E-05		F
6	0.0052	2.0E-04	5.5E-04	2.1E-05		F

TABLE 15

Comments and Observations
Press drop to 92: increased flow to 150 to bring press up to 250: coatings, black film
Press drop to 350, rose slowly and fluctuated: thickness monitor working
Press drop to 97 and stayed: when flow increased to 150, press increased to 500: trouble matching power
Press drop to 227, rose and fluctuated
Press drop to 209, rose, and fluctuated
Press fluctuated: film, dark purple, black

TABLE 15

Scratch 1	Scratch 2	Pull psi	Acid CuSO4 5cry	10cry	Neut CuSO4 5cry	10cry	Ferroxil 5cry	10cry	Salt Spray. h	Conductance µS
2000	3000	720								
9000		1850								
			13.8	13.8	11.4	12.7	4.3	9.2		
									128	
										3
10	1000	410								
			5.6	7.2	0.4	0.6	0.2	0.4		
										55
									32	
300	10000	10								
700	10000	860								
			1.4	3.2	1.0	1.6	0.4	1.7		
										3
									72	
10	8000									
2000	10000	50								
			6.6	8.6	0.9	1.5	7.5	11.7		
										20
									120	
5000	10000	650								
			0.7	4.5	2.6	8.3	5.5	12.8		
										57
									72	
9000	10000	20								
		0								
			0.5	3.2	0.5	4.8	19.2	38.3		
									24	
										20

TABLE 15

Abrasion ml. sand
>5000
600
200
400
200
400

TABLE 15

Notes and Observations
Pull (in exc of). small amt: scratch. film flakes
Pull (load to failure). no film left
Ro CuSO4. 1 cry 9m 30s: both CuSO4. crop dried
Salt test failed 128h @100%(test interrupted 2X)
Black film makes salt test (B93-5) hard to judge
Pull test (in excess of), some film left
Ferroxil. copper cry. then blue(2:33.12:01)
Salt test failed 32 h @ 95% (test interrupted)
Pull test (in excess of). some film left
Pull test same; scratch >10000 (max)
Salt test failed 72 h @95% (test interrupted)
Pull (in exc of). some left: scratch >10000 (max)
Salt test failed 120 h @95%
Pull (in exc of). small amt; scratch >10000 max
N CuSO4. Ferrox: crop dried, cry around edges
Salt test failed 72h @98% (test interrupted twice)
Pull (in exc of). some left: scratch >10000 (max)
Film flakes off, bare metal spots (all B94's)
Ferroxil. 3 cry after 11m 30s
Salt test failed 24 h @98% (test interrupted)

TABLE 15

Design Order	Run Number	Sample Number	XPS Binding Energies wrt C1s at 284.7				
			C1s chg	O1s	S2p	N1s	C1s
7	A59A87	4	1.7	532.2	163.7		0.434
3	A59B96	4	0.4	532.0	164.1/168	399.3	0.437
6	A59B95	2	1.4	531.9	163.0		0.440

TABLE 15

XPS Atomic Fractions

O1s	S2p	N1s
0.419	0.147	
0.425	0.047	0.091
0.375	0.185	

TABLE 16

DATA FOR HMDSO DESIGN I

Design Order	Run Number	Power, w set	Power, w obt	Flow set	Flow sccm	Press set	Temp. degC set	Temp. degC obt	Time min
1	A59S0128	50		15	3.5	350	150	147	30
									30
									30
									30
									30
									30
2	A59S0124	200	196	15	3.5	350	250	242	30
									30
									30
									30
									30
									30
3	A59S0123	50		150	30	350	250	251	30
									30
									30
									30
									30
									30
4	A59S0129	200	160	150	30	350	150	181	30
									30
									30
									30
									30
									30
5	A59S0130	50		15	3.5	1000	250	250	30
									30
									30
									30
									30
									30
6	A59S0126	200	187	15	3.5	1000	150	155	30
									30
									30
									30
									30
									30
7	A59S0127	50		150	30	1000	150	159	30
									30
									30
									30
									30
									30
8	A59S0125	200	154	150	30	1000	250	263	30
									30
									30
									30
									30
									30

TABLE 16

Sample Number	Weight Gain, g	Wt Rate g/min	Thickness g/sqcm	Rate g/sqcm/min	Quality
1	0.0013	4.3E-05	1.4E-04	4.5E-06	F
2	0.0011	3.7E-05	1.1E-04	3.8E-06	F
3	0.0007	2.3E-05	1.1E-04	3.7E-06	F
4	0.0006	2.0E-05	9.6E-05	3.2E-06	F
5	0.0003	1.0E-05	8.4E-05	2.8E-06	F
6	0.0004	1.3E-05	1.1E-04	3.7E-06	F
1	0.0018	6.0E-05	1.9E-04	6.2E-06	F
2	0.0023	7.7E-05	2.4E-04	7.9E-06	F
3	0.0012	4.0E-05	1.9E-04	6.4E-06	F
4	0.0013	4.3E-05	2.1E-04	6.9E-06	F
5	0.0007	2.3E-05	2.0E-04	6.5E-06	F
6	0.0005	1.7E-05	1.4E-04	4.6E-06	F
1	0.0000				F
2	0.0001	3.3E-06	1.0E-05	3.4E-07	F
3	0.0000				F
4	0.0000				F
5	0.0001	3.3E-06	2.8E-05	9.2E-07	F
6	-0.0002				F
1	0.0029	9.7E-05	3.0E-04	1.0E-05	F
2	0.0029	9.7E-05	3.0E-04	1.0E-05	F
3	0.0020	6.7E-05	3.3E-04	1.1E-05	F
4	0.0022	7.3E-05	3.6E-04	1.2E-05	F
5	0.0011	3.7E-05	3.0E-04	1.0E-05	F
6	0.0012	4.0E-05	3.3E-04	1.1E-05	F
1	0.0008	2.7E-05	8.4E-05	2.8E-06	F
2	0.0011	3.7E-05	1.1E-04	3.8E-06	F
3	0.0007	2.3E-05	1.1E-04	3.7E-06	F
4	0.0007	2.3E-05	1.1E-04	3.7E-06	F
5	0.0006	2.0E-05	1.6E-04	5.5E-06	F
6	0.0003	1.0E-05	8.4E-05	2.8E-06	F
1	0.0036	1.2E-04	3.6E-04	1.2E-05	F
2	0.0024	8.0E-05	2.5E-04	8.3E-06	F
3	0.0015	5.0E-05	2.4E-04	8.0E-06	F
4	0.0012	4.0E-05	1.9E-04	6.4E-06	F
5	0.0006	2.0E-05	1.6E-04	5.5E-06	F
6	0.0008	2.7E-05	2.2E-04	7.4E-06	F
1	0.0034	1.1E-04	3.6E-04	1.2E-05	F
2	0.0034	1.1E-04	3.6E-04	1.2E-05	F
3	0.0024	8.0E-05	3.9E-04	1.3E-05	F
4	0.0033	1.1E-04	5.4E-04	1.8E-05	F
5	0.0016	5.3E-05	4.5E-04	1.5E-05	F
6	0.0010	3.3E-05	2.8E-04	9.2E-06	F
1	0.0008	2.7E-05	8.4E-05	2.8E-06	F
2	0.0015	5.0E-05	1.6E-04	5.2E-06	F
3	0.0007	2.3E-05	1.1E-04	3.7E-06	F
4	0.0069	2.3E-04	1.1E-03	3.7E-05	F
5	0.0002	6.7E-06	5.7E-05	1.9E-06	F
6	0.0007	2.3E-05	2.0E-04	6.5E-06	F

TABLE 16

Comments and Observations
Film, rainbow: Press fluctuates between 334-363, then lines out; Salt test (1) failed @97%; Pull (3) 5% film left; Micro. (4) CuSO4, very small Cu crys; Ferroxil, blue crys
Film, rainbow: Salt (1) failed @100%; Pull (4) 60% film left; Micro. (3) CuSO4, Cu crys; Ferroxil, blue crys
Film, rainbow; Pull (3) stud not affix to sample; Micro. (4) CuSO4, Cu crys; Ferroxil, blue crys; Salt (2) failed @95-96%
Film, silver with faint lime rainbow; Flow needed periodic adjustment; Salt (1) failed @95%; Pull (3) 20% film left?; Micro. (4) CuSO4, brown, small Cu crys; Ferroxil, blue crys, 100+ faint, small brown spots
Film, rainbow; Press fluctuated between 976-1020 continuously; Salt (1) failed @98%; Pull (3) 30-40% film left; Micro. (4) CuSO4, Cu, gold, silver, brown crys; Ferroxil, blue crys
Film, silver, transparent & peels around edges; temp mon failed after 10 min; Salt (2) failed @95% Pull (3) stud not affix to sample; Micro. (4) within sec large area turns Cu from CuSO4, blue from Ferroxil, film wrinkles; film can be easily scraped off with needle
Film, silver with white sticky, powder-like surface; Salt (1) failed @98%; Pull (4) 99% film left; Micro. (3) CuSO4, gold, Cu, rust crys; Ferroxil, blue crys, soln tends to roll on sample surface
Film, rainbow w gold hue; changed temp mon 12 min into run; white foam on samples; Salt (2) failed @95%; Pull (3) stud came off; Micro. (4) CuSO4, Cu crys (Neut; small crys, also brown, rust); Ferroxil, blue crys

<u>TABLE 16</u>

Scratch 1	Scratch 2	Pull psi	Salt h	Acid 5cry	CuSO4 10cry	Neut 5cry	CuSO4 10cry	Ferroxil 5cry	10cry	Conductance μS	Abrasi… ml sand
4	700	0	80	0.3	0.8	0.4	0.7	2.0	8.4	580	30
20	400	90	48	0.4	0.5	0.4	0.6	2.6	5.1	34	40
4	40		120	0.1	0.1	0.2	0.2	4.2	9.5	200	40
100	500	140	144	3.8	5.1	0.4	1.6	15.0		9	20
4	500	190	144	1.2	1.9	0.3	0.8	7.0	15.0	700	40
50	1000		6							2100	
300	700	410	98	0.5	1.0	0.9	1.6	0.4	0.8	310	40
4	1000		56	0.3	0.6	0.4	0.7	0.2	0.4	456	40

TABLE 16

Design Order	Run Number	Power, w set	obt	Flow set	Flow sccm	Press set	Temp, degC set	obt	Time min
0	A59SO120	95		60	7	675	200	161	30
									30
									30
									30
									30
									30
0	A59SO121	95		80	7	675	200	205	30
									30
									30
									30
									30
									30
0	A59SO122	95		90	8	675	200	204	30
									30
									30
									30
									30
									30
0	A59SO131	95		90	8	675	200	204	30
									30
									30
									30
									30
									30
0	A59SO132	95		90	8	675	200	200	30
									30
									30
									30
									30
									30

TABLE 16

Sample Number	Weight Gain, g	Wt Rate g/min	Thickness g/sqcm	Rate g/sqcm/min	Quality
1	0.0022	7.3E-05	2.3E-04	7.6E-06	F
2	0.0021	7.0E-05	2.2E-04	7.2E-06	F
3	0.0015	5.0E-05	2.4E-04	8.0E-06	F
4	0.0013	4.3E-05	2.1E-04	6.9E-06	F
5	0.0006	2.0E-05	1.6E-04	5.5E-06	F
6	0.0007	2.3E-05	2.0E-04	6.5E-06	F
1	0.0017	5.7E-05	1.8E-04	5.9E-06	F
2	0.0012	4.0E-05	1.2E-04	4.1E-06	F
3	0.0011	3.7E-05	1.8E-04	5.9E-06	F
4	0.0010	3.3E-05	1.6E-04	5.3E-06	F
5	0.0005	1.7E-05	1.4E-04	4.6E-06	F
6	0.0005	1.7E-05	1.4E-04	4.6E-06	F
1	0.0023	7.7E-05	2.4E-04	7.9E-06	F
2	0.0013	4.3E-05	1.4E-04	4.5E-06	F
3	0.0005	1.7E-05	8.1E-05	2.7E-06	F
4	0.0016	5.3E-05	2.6E-04	8.5E-06	F
5	0.0002	6.7E-06	5.7E-05	1.9E-06	F
6	0.0002	6.7E-06	5.7E-05	1.9E-06	F
1	0.0027	9.0E-05	2.8E-04	9.3E-06	F
2	0.0041	1.4E-04	4.2E-04	1.4E-05	F
3	0.0017	5.7E-05	2.7E-04	9.1E-06	F
4	0.0044	1.5E-04	7.2E-04	2.4E-05	F
5	0.0019	6.3E-05	5.4E-04	1.8E-05	F
6	0.0014	4.7E-05	3.9E-04	1.3E-05	F
1	0.0020	6.7E-05	2.1E-04	6.9E-06	F
2	0.0023	7.7E-05	2.4E-04	7.9E-06	F
3	0.0010	3.3E-05	1.6E-04	5.3E-06	F
4	0.0014	4.7E-05	2.2E-04	7.5E-06	F
5	0.0008	2.7E-05	2.2E-04	7.4E-06	F
6	0.0007	2.3E-05	2.0E-04	6.5E-06	F

TABLE 16

Sample Number	Weight Gain,g	Wt Rate g/min	Thickness g/sqcm	Rate g/sqcm/min	Qual ity
1	0.0022	7.3E-05	2.3E-04	7.6E-06	F
2	0.0021	7.0E-05	2.2E-04	7.2E-06	F
3	0.0015	5.0E-05	2.4E-04	8.0E-06	F
4	0.0013	4.3E-05	2.1E-04	6.9E-06	F
5	0.0006	2.0E-05	1.6E-04	5.5E-06	F
6	0.0007	2.3E-05	2.0E-04	6.5E-06	F
1	0.0017	5.7E-05	1.8E-04	5.9E-06	F
2	0.0012	4.0E-05	1.2E-04	4.1E-06	F
3	0.0011	3.7E-05	1.8E-04	5.9E-06	F
4	0.0010	3.3E-05	1.6E-04	5.3E-06	F
5	0.0005	1.7E-05	1.4E-04	4.6E-06	F
6	0.0005	1.7E-05	1.4E-04	4.6E-06	F
1	0.0023	7.7E-05	2.4E-04	7.9E-06	F
2	0.0013	4.3E-05	1.4E-04	4.5E-06	F
3	0.0005	1.7E-05	8.1E-05	2.7E-06	F
4	0.0016	5.3E-05	2.6E-04	8.5E-06	F
5	0.0002	6.7E-06	5.7E-05	1.9E-06	F
6	0.0002	6.7E-06	5.7E-05	1.9E-06	F
1	0.0027	9.0E-05	2.8E-04	9.3E-06	F
2	0.0041	1.4E-04	4.2E-04	1.4E-05	F
3	0.0017	5.7E-05	2.7E-04	9.1E-06	F
4	0.0044	1.5E-04	7.2E-04	2.4E-05	F
5	0.0019	6.3E-05	5.4E-04	1.8E-05	F
6	0.0014	4.7E-05	3.9E-04	1.3E-05	F
1	0.0020	6.7E-05	2.1E-04	6.9E-06	F
2	0.0023	7.7E-05	2.4E-04	7.9E-06	F
3	0.0010	3.3E-05	1.6E-04	5.3E-06	F
4	0.0014	4.7E-05	2.2E-04	7.5E-06	F
5	0.0008	2.7E-05	2.2E-04	7.4E-06	F
6	0.0007	2.3E-05	2.0E-04	6.5E-06	F

TABLE 16

Comments and Observations
Film, rainbow w gray hue; temp kept decreasing thru run; Salt (1) failed @98%; Pull (3) 80% film left; Micro. (4) CuSO4, Cu crys. within 45 sec brown streaks cover 30-40% of drop; Ferr, blue crys. within 5 sec brown streaks cover 10-60% drop, brown spots form 5 & 10 crys in 0.1 min
Film, rainbow w gray hue; flow @ 90; Salt (1) failed @98%; Pull (3) stud came off; Micro. (4) CuSO4, Cu crys; Ferroxil, blue crys
Film, rainbow w gray hue; Pull (3) stud came off; Micro. (4) CuSO4, Cu & small silver crys; Ferroxil, blue crys, only 5 formed; Salt (1) failed @95%; salt spray sample not crusted with rust like other samples in this matrix series
Film, silver w faint lime rainbow; Salt (2) failed @98%; Pull (3) 80% film left; Micro. (4) CuSO4, Cu, silver, rust spots; Ferroxil, blue crys
Film, silver w faint lime rainbow; Salt (1) failed @98%; Pull (3) 10 or 90% film left?; Micro. (4) CuSO4, very small silver crys, a couple rust, Cu ones; Ferroxil, blue crys (1 in 25 min)

TABLE 16

Scratch 1	Scratch 2	Pull psi	Salt h	Acid 5cry	CuSO4 10cry	Neut 5cry	CuSO4 10cry	Ferroxil 5cry	Ferroxil 10cry	Conductance μS	Poros ml san
4	1000	0	48	0.1	0.2	0.1	0.1	0.9	1.8	35	40
4	50		72	0.2	0.4	0.6	0.8	7.1	12.9	930	40
4	1300		168	0.7	1.1	0.4	0.6	14.1		240	30
10	300	20	144	0.3	0.6	0.3	1.8	19.0		20	20
20	500	30	152	0.3	0.7	0.7	1.5			350	30

TABLE 17
DATA FOR HMDSN DESIGN I

Std Ord	Run Number	Power set	Power obt	Flow HMDSN	Flow O2	Press set	Press obt	Time min	Sample Number	Weight Gain, g	Rate g/sqcm/min	Quality
1	86428	50		15	2	250		15	A	5E-04	5.3E-06	F
								15	B	6E-04	6.4E-06	F
								15	C	8E-04	8.5E-06	F
								15	D	1.0E-03	1.1E-05	F
								15	E	4E-04	4.3E-06	F
								15	F	8E-04	8.5E-06	F
2	86414	200		15	2	1000		15	A	2.0E-03	2.1E-05	F
								15	B	2.2E-03	2.4E-05	F
								15	C	2.5E-03	2.7E-05	F
								15	D	1.7E-03	1.8E-05	F
								15	E	1.6E-03	1.7E-05	F
								15	F	1.7E-03	1.8E-05	F
3	86413	50		150	2	1000		15	A	9E-04	9.6E-06	F
								15	B	7E-04	7.5E-06	F
								15	C	1.0E-03	1.1E-05	F
								15	D	1.7E-03	1.8E-05	F
								15	E	1.8E-03	1.9E-05	F
								15	F	1.8E-03	1.9E-05	F
4	86429	200	160	150	2	250	305	15	A	1.5E-03	1.6E-05	F
								15	B	1.7E-03	1.8E-05	F
								15	C	2.2E-03	2.4E-05	F
								15	D	2.7E-03	2.9E-05	F
								15	E	2.4E-03	2.6E-05	F
								15	F	2.7E-03	2.9E-05	F
5	864210	50		15	5	1000		15	A	1.6E-03	1.7E-05	F
								15	B	1.4E-03	1.5E-05	F
								15	C	1.6E-03	1.7E-05	F
								15	D	1.8E-03	1.9E-05	F
								15	E	1.8E-03	1.9E-05	F
								15	F	2.0E-03	2.1E-05	F
6	86416	200	170	15	5	250		15	A	6E-04	6.4E-06	F
								15	B	9E-04	9.6E-06	F
								15	C	1.0E-03	1.1E-05	F
								15	D	1.1E-03	1.2E-05	F
								15	E	8E-04	8.5E-06	F
								15	F	1.0E-03	1.1E-05	F
7	86417	50		150	5	250	300	15	A	1.4E-03	1.5E-05	F
								15	B	1.1E-03	1.2E-05	F
								15	C	1.4E-03	1.5E-05	F
								15	D	2.3E-03	2.4E-05	F
								15	E	1.8E-03	1.9E-05	F
								15	F	1.9E-03	2.0E-05	F
8	86415	200	147	150	5	1000		15	A	2.5E-03	2.7E-05	F
								15	B	4.7E-03	5.0E-05	F
								15	C	4.1E-03	4.4E-05	F
								15	D	4.0E-03	4.3E-05	F
								15	E	3.9E-03	4.2E-05	F
								15	F	4.1E-03	4.4E-05	F
0	86381	125		90	3	625		30	A	5.2E-03	2.8E-05	F
								30	B	5.7E-03	3.0E-05	F
								30	C	3.3E-03	1.8E-05	F
								30	D	5.0E-03	2.7E-05	F
								30	E	4.9E-03	2.6E-05	F

TABLE 17

								30	F	4.9E-03	2.6E-05	F
0	86382	125		90	3	625		15	A	1.7E-03	1.8E-05	F
								15	B	2.2E-03	2.4E-05	F
								15	C	2.8E-03	3.0E-05	F
								15	D	2.9E-03	3.1E-05	F
								15	E	2.8E-03	3.0E-05	F
								15	F	2.6E-03	2.8E-05	F
0	864211	125		90	3	625		15	A	1.8E-03	1.9E-05	F
								15	B	1.7E-03	1.8E-05	F
								15	C	1.6E-03	1.7E-05	F
								15	D	2.8E-03	3.0E-05	F
								15	E	2.6E-03	2.8E-05	F
								15	F	2.5E-03	2.7E-05	F
0	864212	125		90	3	625		15	A	1.9E-03	2.0E-05	F
								15	B	2.0E-03	2.1E-05	F
								15	C	1.8E-03	1.9E-05	F
								15	D	2.9E-03	3.1E-05	F
								15	E	2.9E-03	3.1E-05	F
								15	F	2.4E-03	2.6E-05	F
1	864213	50		15	2	250		15	A	9E-04	9.6E-06	F
								15	B	7E-04	7.5E-06	F
								15	C	8E-04	8.5E-06	F
								15	D	1.2E-03	1.3E-05	F
								15	E	1.1E-03	1.2E-05	F
								15	F	1.0E-03	1.1E-05	F
	864214	200	183	15	2	172	185	30	A	3.1E-03	1.6E-05	F
								30	B	1.6E-03	8.5E-06	F
								30	C	3.5E-03	1.9E-05	F
								30	D	7E-04	3.7E-06	F
								30	E	9E-04	4.8E-06	F
								30	F	1.3E-03	6.9E-06	F
	864315	200	179	15	2	196	216	35	A	9E-04	4.1E-06	F
								35	B	1.1E-03	5.0E-06	F
								35	C	9E-04	4.1E-06	F
								35	D	3.2E-03	1.5E-05	F
								35	E	3.6E-03	1.6E-05	F
								35	F	3.0E-03	1.4E-05	F
	864316	200	181	10	2	188	188	60	A	4.5E-03	1.2E-05	F
								60	B	4.0E-03	1.1E-05	F
								60	C	3.7E-03	9.9E-06	F
								60	D	1.1E-03	2.9E-06	F
								60	E	9E-04	2.4E-06	F
								60	F	1.2E-03	3.2E-06	F

TABLE 17

Glass Stage	Comments and Observations	Scratch 1	Scratch 2	Pull psi
G	Film rainbow; rotating stage			
G	not running; Pull B&E stud	4	2000	
G	came off, 0-5% film left on			
S	sample			
S		20	2000	
S				
G	Film on glass silver w aqua	4	1500	
G	rainbow, on stage silver &			
G	uneven film, easily scratch;			
S	Pull A&E stud came off, 0-20%			
S	film left	20	2000	
S				
G	Film on glass silver, rainbow,			
G	on stage silver, oily; Pull C&			
G	E 80-100% film left, Pull F	4	100	20
S	not wiped w TCE, stud came off			
S		4	4	780
S		4	4	
G	Film dull gray; Trouble			
G	setting power; Pull B 95% film	4	400	190
G	left, Pull D stud came off,			
S	90% film left	800	1500	
S				
S				
G	Film slight lime color; Pull	50	4000	
G	A stud came off, Pull E 10%			
G	film left			
S				
S		150	4000	20
S				
G	Film green & pink rainbow;			
G	Trouble matching power; Pull C			
G	20-30% film left, Pull E stud	4	500	20
S	came off, 30% film left			
S		10	300	
S				
G	Film on glass slight rainbow,			
G	on stage silver; Pull B 60-70%	4	300	50
G	film left, Pull D stud came			
S	off	10	800	
S				
S				
G	Film dull gray; Trouble			
G	matching power; Pull B&D stud	4	1500	
G	came off, 90-100% film left			
S		30	1000	
S				
S				
G	Film silver; Pull B 70-80%			
G	film left, Pull F 10% film	200	900	20
G	left			
S				
S				

TABLE 17

S		500	700	450
G	Film silver; Pull B 20% blue			
G	film left, Pull E 10% blue	4	1000	80
G	film left			
S				
S		500	1500	0
S				
G	Film on glass slightly lime			
G	colored rainbow, on stage dull	100	500	0
G	gray; Pull B 10% blue film			
S	left, Pull E stud came off,			
S	1-5% film left	400	800	
S				
G	Film on glass slight lime			
G	color, on stage dull gray;	500	700	
G	Pull B stud came off, 20% blue			
S	film left, Pull D stud came	400	500	
S	off, 95% film left			
S				
G	Repeat Std Ord 1; film			
G	rainbow; Pull B 60% film left,	4	2500	120
G	Pull E 10-20% film left			
S				
S		4	2000	450
S				
G	Film on glass crazed, slight			
G	lime rainbow, on stage flaky			
G	rainbow; Press open; Pull C 0%	10	500	440
S	film left, Pull E 60% film			
S	left, both Pulls cream film	4	90	120
S	peels off			
G	Repeat run 864214; film flaky			
G	rainbow; Press open; stage not			
G	at ground potential; Pull C	4	500	2920
S	30% film left, Pull F 60% film			
S	left, film similar to 864214,			
S	samples D-F more flaky film	4	20	2920
G	Film rainbow; Press open;	40	900	100
G	Pull A 5% film left; Pull D			
G	stud came off			
S		10	2000	
S				
S				

TABLE 18

DATA FOR HMDSN DESIGN II

Std Ord	Run Number	Power set	Power obt	Flow HMDSN	Flow O2	Press set	Press obt	Time min	Sample Number	Weight Gain, g	Rate g/sqcm/min	Quality
1	86445	50		10	0	175		60	A	1.0E-03	2.7E-06	F
								60	B	1.0E-03	2.7E-06	F
								60	C	1.0E-03	2.7E-06	F
								60	D	1.2E-03	3.2E-06	F
								60	E	1.1E-03	2.9E-06	F
								60	F	9E-04	2.4E-06	F
2	86457	200	160	10	0	250		60	A	2.8E-03	7.5E-06	F
								60	B	3.0E-03	8.0E-06	F
								60	C	4.2E-03	1.1E-05	F
								60	D	5E-04	1.3E-06	F
								60	E	6E-04	1.6E-06	F
								60	F	5E-04	1.3E-06	F
3	86446	50		15	0	250		60	A	1.9E-03	5.1E-06	F
								60	B	1.9E-03	5.1E-06	F
								60	C	2.0E-03	5.3E-06	F
								60	D	2.2E-03	5.9E-06	F
								60	E	2.2E-03	5.9E-06	F
								60	F	2.3E-03	6.1E-06	F
4	864510	200	178	15	0	175	198	60	A	5.1E-03	1.4E-05	F
								60	B	4.5E-03	1.2E-05	F
								60	C	5.4E-03	1.4E-05	F
								60	D	1.6E-03	4.3E-06	F
								60	E	1.8E-03	4.8E-06	F
								60	F	1.8E-03	4.8E-06	F
5	86459	50		10	2	250		60	A	1.1E-03	2.9E-06	F
								60	B	1.2E-03	3.2E-06	F
								60	C	1.1E-03	2.9E-06	F
								60	D	1.1E-03	2.9E-06	F
								60	E	1.1E-03	2.9E-06	F
								60	F	1.1E-03	2.9E-06	F
6	86443	200	169	10	2	175	183	30	A	6E-04	3.2E-06	F
								30	B	7E-04	3.7E-06	F
								30	C	7E-04	3.7E-06	F
								30	D	6E-04	3.2E-06	F
								30	E	5E-04	2.7E-06	F
								30	F	5E-04	2.7E-06	F
6	864813	200	169	10	2	175	194	60	A	4.4E-03	1.2E-05	F
								60	B	3.4E-03	9.1E-06	F
								60	C	4.1E-03	1.1E-05	F
								60	D	1.2E-03	3.2E-06	F
								60	E	1.2E-03	3.2E-06	F
								60	F	1.4E-03	3.7E-06	F
7	86444	50		15	2	175	191	60	A	2.5E-03	6.7E-06	F
								60	B	2.5E-03	6.7E-06	F
								60	C	2.3E-03	6.1E-06	F
								60	D	2.5E-03	6.7E-06	F
								60	E	2.4E-03	6.4E-06	F
								60	F	2.4E-03	6.4E-06	F
8	86458	200	179	15	2	250		60	A	3.5E-03	9.3E-06	F
								60	B	2.8E-03	7.5E-06	F
								60	C	3.4E-03	9.1E-06	F
								60	D	1.1E-03	2.9E-06	F
								60	E	1.0E-03	2.7E-06	F

TABLE 18

						60	F	1.0E-03	2.7E-06	F
0	86431	125	12.5	2	213	60	A	3.5E-03	9.3E-06	F
						60	B	4.8E-03	1.3E-05	F
						60	C	4.5E-03	1.2E-05	F
						60	D	2.2E-03	5.9E-06	F
						60	E	2.4E-03	6.4E-06	F
						60	F	2.1E-03	5.6E-06	F
0	86442	125	12.5	2	213	60	A	2.1E-03	5.6E-06	F
						60	B	1.9E-03	5.1E-06	F
						60	C	1.4E-03	3.7E-06	F
						60	D	1.6E-03	4.3E-06	F
						60	E	2.0E-03	5.3E-06	F
						60	F	1.2E-03	3.2E-06	F
0	864511	125	12.5	2	213	60	A	4.4E-03	1.2E-05	F
						60	B	2.2E-03	5.9E-06	F
						60	C	4.6E-03	1.2E-05	F
						60	D	2.0E-03	5.3E-06	F
						60	E	2.1E-03	5.6E-06	F
						60	F	2.3E-03	6.1E-06	F
0	864512	125	12.5	2	213	60	A	4.9E-03	1.3E-05	F
						60	B	4.6E-03	1.2E-05	F
						60	C	4.3E-03	1.2E-05	F
						60	D	2.4E-03	6.4E-06	F
						60	E	2.3E-03	6.1E-06	F
						60	F	2.3E-03	6.1E-06	F

TABLE 18

Glass Stage	Comments and Observations	Scratch 1	Scratch 2	Pull psi
G	Film on glass rainbow, flaky;			
G	on stage more flaky, silver			
G	Pull(C)30% film left, may have	4	100	9280
S	been loaded improperly, Pull			
S	(F) 70% film left			
S		4	40	2570
G	Film rainbow streaky, flaky	4	30	150
G	gold; Pull(A) 95% film left,			
G	Pull(D) 5% film left			
S		4	30	1380
S				
S				
G	Film silver w slight lime	200	400	410
G	rainbow; Pull(A)95% film left,			
G	Pull(D) stud came off			
S		90	400	
S				
S				
G	Film on glass gold w pink			
G	stripes; on stage gold, crazed	4	700	8810
G	& flaky, silver underneath;			
S	Pull(B) 90% film left, Pull(E)			
S	50% film left	20	600	7760
S				
G	Film rainbow; Pull(C) no film			
G	left, Pull(E) 50% film left			
G		4	500	10
S				
S		4	1000	0
S				
G	Film green, pink rainbow;			
G	Pull(B,E) stud came off	4	100	
G				
S				
S		4	800	
S				
G	Repeat; film on glass pink,			
G	green rainbow; on stage same			
G	w gold, crazed around edges;	4	300	
S	Pull(C,F) stud came off, no			
S	film left			
S		10	300	
G	Film silver w very slight lime	100	500	
G	rainbow; Pull(A,D) stud came			
G	off, 0-10% film left			
S		100	800	
S				
S				
G	Film on glass rainbow, silver	4	10	2750
G	where coat flaked; on stage			
G	silver, more flaky coat;			
S	Scratch 2(A)300 on rainbow,	20	2000	830
S	1/4 of sample; Pull(A,D) 40%			

TABLE 18

S	film left			
G	Film on glass silver, rainbow;	10	500	460
G	on stage pale gold, crazed,			
G	flaky; Pull(A) 20% film left,			
S	Pull(E) 30% film left			
S		40	200	9870
S				
G	Film on glass silver, flaky;	4	400	4920
G	on stage pale gold, flaky;			
G	flaky rainbow when removed			
S	from vacuum; Pull(A) 60% film			
S	left, Pull(E) 30% film left;	4	300	5900
S	Scr2(E)70, 400 opposite edges			
G	Film silver w faint lime	200	3000	
G	rainbow, flaky around edges;			
G	Pull(A) stud came off, Pull(E)			
S	80% film left			
S		4	10	2480
S				
G	Film on glass silver w faint	200	1000	
G	lime rainbow, flaky at edges;			
G	on stage same w gold, crazed,			
S	flaky; Pull(A) stud came off,			
S	no film left, Pull(E) 90% film	4	20	1480
S	left			

TABLE 19

SUMMARY OF AVERAGED DATA OF RUNS IN THIOPHENE
DESIGN I, II, III, AND IV

SAMPLE #	DESIGN ORDER	PWR WATTS	FLOW SCCM	PRESS. mTorr	TEMP °C	TIME min.	FILM QUAL	WT.GN. mg	RATE µg/min	THKNS µg/cm^2	THK.RATE µg/cm^2/min	SCR. 1,g	SCR. 2,g	PULL psi	S.S. hrs	$CuSO_4$ min	FERR min
A59A9	1	10	2.1	250		12	F	0.2	17	32	2.7	50	100			<1	<1
A59A2	2	10	29	250		15	F/P	3.7	245	590	39	1000	5000	20		3.5	<1
A59A4	3	10	2.1	500		40	P	1.7	43	280	7	500	5000	1140		2.5	<1
A59A7	4	10	20	500		29	P	12.5	435	2000	69						
A59A1	5	50	2.1	250		10	F	0.4	38	60	6	200	500	30		<1	<1
A59A6	6	50	29	250		2	F	0.8	375	120	60	1000	5000			<1	<1
A59A8	7	50	2.1	500		45	F	0.4	9	65	1.4	50	900			<1	<1
A59A3	8	50	29	5000		6	P	6.1	1020	965	161					<1	<1
A59B9	1	50	2.1	500		74	F	0.8	10	122	1.6		500			<1	<1
A59B3	2	50	29	500		4	F/P	2.8	950	610	153						
A59B4	3	50	2.1	510		17	P	1.1	67	178	11						
A59B7	4	50	20	1000		7	P	5.6	803	868	124						
A59B2	5	100	2.1	500		25	F	0.4	15	63	3	100	600			<1	<1
A59B6	6	100	29	500		2	F	1.1	540	175	88	10	1000			<1	<1
A59B8	7	100	2.1	1000		42	F	1.3	32	212	5		200			<1	<1
A59B1	8	100	29	1000		3	P	3.6	1200	570	190						
A59C1	5	50	3	150		6	F	0.5	79	75	13	80	500			<1	<1
A59C2	5	100	3	450		10	F	0.4	35	56	6	20	1000			3	<1
A59A13	1	10	3	220		120	F	3.6	60	517	43	2000	9000		1	4.5	5.9
A59A11	5	50	3	153		120	F	2.8	23	368	3.1	900	10000		1	2.6	<1
A59C3	5	50	3	250		38	F	1.1	28	170	4.4	500	5000		48	<1	2.2
A59A10	6	50	29	250		12	F	4.3	357	583	49	500	5000	10	1	1.4	<1
A59C4	7	50	3	400		120	F	2.9	24	380	3.2	500	7000		1	3.3	6.0
A59B13	1	50	3	310		120	F/P	3.9	33	537	4.5		10000	10	1	2.8	4.0
A59B11	5	100	3	500		124	F	2.2	18	297	2.4	50	5000	10	48	1.8	1.7
A59B10	6	100	25	500		13	P	7.6	582	1053	81						
A59B12	7	100	3	1000		120	P	4.7	39	645	5.4	1000	10000	810	5	2.9	3.5
A59A59	1	10	3	248	56	10	F	0.2	20	32	3	4					
A59A57	2	50	3	248	147	3	F	0.2	50	41	14	4					
A59A67	3	10	3	485	146	8	F	0.1	12	16	2	4					
A59A65	4	50	3	500	58	4	F	0.2	37	24	6	4	400				
A56A66	5	10	29	249	148	5	F	0.1	20	16	3	4	10				
A59A55	6	50	29	249	57	3	F	0.7	240	110	33	20	700				
A59A61	7	20	29	500	50	13	F	0.2	23	48	4	4	10				
A59A63	8	50	29	500	160	3	F/P	1.0	335	16	5	10	900				
A59B72	1	50	3	249	146	40	F	0.4	10	6.4	1.6	4	10				
A59B70	2	100	3	249	254	22	F	0.1	5	16	0.7	4					
A59B75	3	50	3	500	246	35	F	0.3	9	49	1.4	4	50				
A59B71	4	100	3	500	156	45	F	0.3	6	41	0.9	4	20				
A59B68	5	50	29	250	240	15	F	1.1	76	190	12.5	40	2500				
'59B74	6	100	29	253	155	15	F	1.6	105	255	17	250	7000				
A59B69	7	50	29	500	140	15	F/P	5.0	330	790	52.5	3000	4500				
A59B73	8	100	29	498	251	15	F	1.8	120	285	19	70	3000				

TABLE 20

SUMMARY OF AVERAGED DATA OF SELECTED RUNS FROM THIOPHENE DESIGNS III AND IV

SMPL. #	DES ORDR	PWR WTTS	FLOW SCCM	PRESS mTorr	TEMP °C	TIME min.	FILM QLTY	WT.GN. mg	RATE µg/ min.	THKNS µg/ cm²	THK RT µg/cm2/ min.	SCR 1.g	SCR 2.g	PULL psi	S.S. hrs.	$CuSO_4$ min	FERR min	COND µS	ABRAS ml/sand	[illegible]
A59A86	1	10	3	248	54	120	F	4.4	36	595	5.0	700	3000	0	126	1.2	9.8	14	200	
A59A85	2	50	3	248	149	36	F	0.4	12	57	1.6	4	20	300	48	<1	3.7	800	700	
A59A90	3	10	3	499	150	120	F	2.8	24	387	3.2	900	8000	1280	152	19.1	17.4	30	300	
A59A88	4	50	3	498	76	83	F	2.7	32	360	4.3	700	3000	1060	245	6.9	20.2	10	200	7
A59A89	5	10	28	248	148	66	F	2.7	41	360	5.5	10	5000	1070	216	3.8	23.7	30	400	7
A59A84	6	50	29	248	68	14	F	3.1	220	420	30	800	7000	160	72	90	1.6	22	200	
A59A87	7	20	29	499	47	125	F/P	4.2	34	562	4.5	90	3000	150	120	23.2	4.5	14	100	
A59B93	1	50	20	230	155	120	F	19.0	159	2533	21	2000	3000	1850	128	13.8	9.2	5	>5000	7
A59B96	3	50	3	500	248	120	F	1.3	886	138	1.2	10	1000	410	32	7.2	<1	55	600	
A59B92	4	100	2	71	134	52	F	3.6	69	490	9.4	700	10000	860	72	3.2	1.7	9	200	
A59B91	5	50	29	250	238	42	F	3.0	70	393	9.4	2000	10000	50	120	8.6	11.7	20	400	7
A59B95	6	100	29	250	151	29	F	4.9	167	657	22.6	5000	10000	650	72	8.3	12.8	57	200	
A59B94	8	100	29	498	252	26	F	4.5	172	608	23.4	9000	10000	20	24	4.8	38.3	20	400	

TABLE 21

SUMMARY OF AVERAGED DATA OF RUNS IN HMDSO DESIGN I

SMPL. #	DES ORDR	PWR WTTS	FLOW SCCM	PRESS mTorr	TEMP ^{0}C	TIME min.	FILM QLTY	WT.GN. mg	RATE μg/min.	THKNS μg/cm^2	THK RT μg/cm2/min.	SCR 1,g	SCR 2,g	PULL psi	S.S. hrs.	$CuSO_4$ min	FERR min	COND μS	ABRAS ml/sand
S0128	1	50	3.5	350	147	30	F	0.7	24	108	3.6	4	700	0	80	<1	8.4	580	200
S0124	2	196	3.5	350	242	30	F	1.3	43	195	6.5	20	400	90	48	<1	9.1	34	400
S0123	3	50	30	350	251	30	F	0.1	3.3	11	0.4	4	40		120	<1	9.5	200	400
S0129	4	160	30	350	181	30	F	2.1	68	320	10.7	100	500	140	144	5.1	15	9	200
S0130	5	50	3.5	1000	250	30	F	0.7	23	109	3.7	4	500	190	144	1.9	15	700	400
S0126	6	187	3.5	1000	155	30	F	1.7	56	237	7.9	50	1000		6			2100	
S0127	7	50	30	1000	159	30	F/P	2.5	84	397	13.2	300	700	410	98	1.6	<1	310	400 1
S0125	8	154	30	1000	263	30	F	1.8	60	285	9.5	4	1000		56	<1	<1	456	400
S0120	M	95	7	675	161	30	F	1.4	47	210	7.0	4	1000		48	<1	1.8	39	400
S0121	M	95	7	675	205	30	F	1.0	34	153	5.1	4	50		72	<1	12.9	930	400
S0122	M	95	8	675	204	30	F	1.0	34	140	4.6	4	1300		168	<1	14.1	240	300
S0131	M	95	8	675	204	30	F	2.7	90	438	14.6	10	300	20	144	<1	19.0	20	200
S0132	M	95	8	675	200	30	F	1.5	51	208	6.9	20	500	30	152	1.5		350	300

TABLE 22

SUMMARY OF AVERAGED DATA OF RUNS IN HMDSN DESIGNS I AND II

SAMPLE #	DESIGN ORDER	PWR WATTS	FLOW HMDSN	SCCM O_2	PRESS. mTorr	TIME min.	FILM QUAL	WT.GN. mg	RATE µg/min	THKNS µg/cm^2	THK.RATE µg/cm^2/min	SCR. 1.g	SCR. 2.g	PULL psi	S.S. hrs
86428	1	50	3.5	2	250	15	F	0.68		109	7.3	20	2000		
864213	1	50	3.5	2	250	15	F	1.0		152	10.1	4	2500	450	
86414	2	200	3.5	2	1000	15	F	2.0		312	20.8	20	2000		
86413	3	50	29	2	1000	15	F	1.3		210	14.5	4	100	780	
86429	4	160	29	2	305	15	F	2.2		352	23.5	800	1500	190	
864210	5	50	3.5	5	1000	15	F	1.7		270	18	150	4000	20	
86416	6	170	3.5	5	250	15	F	0.9		144	9.6	10	300	20	
86417	7	50	29	5	300	15	F	1.7		264	17.6	10	800	50	
86415	8	147	29	5	1000	15	F	3.9		621	41.4	30	1000		
86382	M	125	8	3	625	15	F	2.5		400	26.7	500	1500	80	
864211	M	125	8	3	625	15	F	2.2		348	23.2	400	800		
864212	M	125	8	3	625	15	F	2.3		372	24.8	500	700		
86381	X	125	8	3	625	30	F	4.8	773	25.8	500	700	450		
864214	X	200	3.5	2	185	30	F/F	1.9		296	9.9	10	500	440	
864215	X	200	3.5	2	216	35	F/F	2.1		339	9.7	4	500	2920	
864316	X	200	2.4	2	188	60	F	2.6		411	6.9	10	2000	100	
86445	1	50	2.4	0	175	60	F/F	1.03		166	2.7	4	100	9280	
86457	2	160	2.4	0	250	60	F/F	1.93		309	5.2	4	30	1380	
86446	3	50	3.5	0	250	60	F	2.08		333	5.6	200	400	410	
864510	4	178	3.5	0	198	60	F/C	3.37		539	9.0	4	700	8810	
86459	5	50	2.4	2	250	60	F	1.18		179	3.0	4	1000	10	
864813	6	169	2.4	2	194	60	F/C	2.62		418	7.0	10	300		
86444	7	50	3.5	2	191	60	F	2.43		389	6.5	100	800		
86458	8	179	35	2	250	60	F/F	2.13		341	5.7	20	2000	2750	48
86431	M	125	3.0	2	213	60	F/C	3.25		520	8.7	40	200	9870	
86442	M	125	3.0	2	213	60	F/F	1.70		272	4.5	4	400	4920	
864511	M	125	3.0	2	213	60	F/F	2.93		469	7.8	200	3000	2480	6
864512	M	125	3.0	2	213	60	F/F	3.47		556	93	200	1000	1480	8

TABLE 23

PROPERTIES OF THE 15 BEST CORROSION PROTECTION COATINGS

SAMPLE #	CTG. WT. $\mu g/cm^2$	CTG. RATE $\mu g/cm^2/min$	SCRATCH #1	SCRATCH #2	PULL psi	SALT SPRAY hrs.	$CuSO_4$ min	FERR. min	COND. μS	ABRASION ml sand
CRITERIA		10	400	1000	100	100				400
A59A88	360	4.3	700	3000	1060	245	6.9	20.2	10	200
A59A89	360	5.5	10	5000	1070	216	3.8	23.7	30	400
A59A90	387	3.2	900	8000	1280	152	19.1	17.4	30	300
A59B93	2533	21	2000	3000	1850	128	13.8	9.2	5	5000
A59A87	562	4.5	90	3000	150	120	23.2	4.5	14	100
A59B91	393	9.4	2000	10000	50	120	8.6	11.7	20	400
A59A84	420	30	800	7000	160	72	90	1.6	22	200
A59B95	657	22.6	5000	10000	650	72	8.3	12.8	57	200
A59B92	490	9.4	700	10000	860	72	3.2	1.7	9	200
A59S0132	208	6.9	20	500	30	152	1.5		350	300
A59S0122	140	4.6	4	1300	0	168	<1	14	240	300
A59S0129	320	10.7	100	500	140	144	5.1	15	9	200
A59S0130	109	3.7	4	500	190	144	1.9	15	700	400
A59S0131	438	14.6	10	300	20	144	<1	19	20	200
A59S0127	397	13.2	300	700	410	98	1.6	<1	310	400

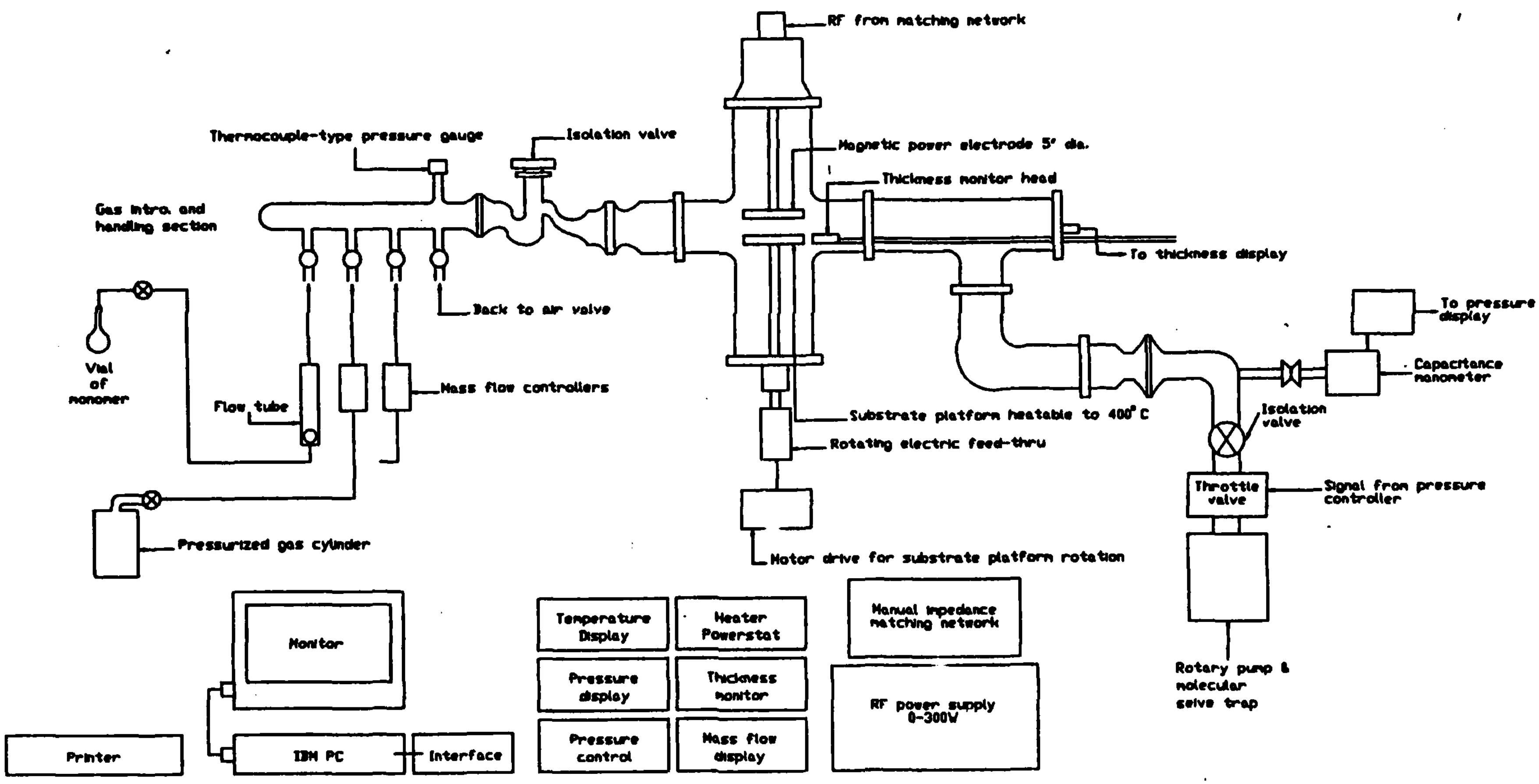
TABLE 24

FIRST ORDER EFFECTS OF PROCESS VARIABLES

					FIRST ORDER EFFECT ON RESPONSES								
DESIGN NO.	VAR	DESN VAR	VAR UNITS	VAR RANGE	THCK RATE	SCR1	SCR2	PULL	SALT SPRAY	$CuSO_4$	FERR SOL	COND	ABRAS
THIOPH I	POWER	C	WATTS	10/50	27.7	-75	-925	-282		-1.275	-0.15		
	FLOW	A	SCCM	2.1/29	78	300	875	-288		0.125	0		
	PRESS	B	mT	250/500	32	-425	-1175	272		-0.325	-0.05		
THIOPH II	POWER	C	WATTS	50/100	25.8	27.5	450			0.23	0.10		
	FLOW	A	SCCM	2.1/29	107	-22.5	50			-0.28	-0.05		
	PRESS	B	mT	500/1000	-5.6	-27.5	-350			-0.28	-0.10		
THIOPH III	POWER	A	WATTS	10/50	11.5	5.5	495						
	FLOW	C	SCCM	3/29	5	5.5	302						
	PRESS	B	mT	250/500	-9	-2.5	149						
	TEMP	D	°C	50/150	-5.5	-2.5	-49						
THIOPH IV	POWER	A	WATTS	50/100	-7.6	-680	741						
	FLOW	C	SCCM	3/29	24.1	836	4229						
	PRESS	B	mT	250/500	10.5	695	-486						
	TEMP	D	°C	150/250	-9.6	-785	-1494						
THIOPH III-A	POWER	A	WATTS	10/100	6.4	76	-1245	-102	-27	17.7	-5.8	219	100
	FLOW	C	SCCM	3/29	8.4	-225	745	-172	-6	-27.6	-1.5	-164	-100
	PRESS	B	mT	250/500	-5.6	169	1245	384	49	-6.2	4.4	-170	-150
	TEMP	D	°C	50/250	-6.5	-219	225	463	-2	-19.3	4.1	233	250
HMDSO I	POWER	A	WATTS	50/200	3.4	-34.5	240	-92.5	-47	0.48	-2.3	202	-100
	FLOW	B	SCCM	3.5/30	3.0	82.5	-90	67.5	35	1.08	-1.7	-610	100
	PRESS	C	mT	350/1000	3.3	57.5	390	92.5	-22	-0.63	-6.5	686	0
	TEMP	D	°C	150/250	-3.8	-105.5	-240	-67.5	10	-1.03	2.5	-402	200
HMDSN I	POWER	A	WATTS	50/200	9.1	172	-525	-216					
	FLOW	B	SCCM	3.5/29	10.0	164	-1225	189					
	OXYGEN	C	SCCM	2/5	4.8	-159	125	-276					
	PRESS	D	mT	250/1000	8.8	-157	625	79					
HMDSN II	POWER	A	WATTS	50/180	2.3	-67.5	183	810					
	FLOW	B	SCCM	2.4/3.5	2.2	75.5	618	325					
	OXYGEN	C	SCCM	0/2	-0.1	-19.5	718	-4280					
	PRESS	D	mT	175/250	-1.4	27.5	383	-3385					

FIGURE 1

VACUUM PLASMA POLYMERIZATION REACTOR AND SYSTEM



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